Tom Delaval.

Access DB# 121537

SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Sabiha Qazi Examiner #: 74141 Date: 5/7/04

Art Unit: 1616 Phone Number 30[illegible] Serial Number: 09/335,025

Mail Box and Bldg Room Location: Results Format Preferred (circle): PAPER DISK E-MAIL

4C70 Rm 4A45

If more than one search is submitted, please prioritize searches in order of need. MEJ

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for Producing Vit. D_3 + Pre Vit D_3

Inventors (please provide full names): Monika Johannsen

Earliest Priority Filing Date: 6/17/1999

For Sequence Searches Only Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.

Please Search for the Process of isolation of Vit D_3 or pre Vit. D_3 or Steroid by as in Cls 1-6

1) Please note normal phase column chromatography] can be Search on Silica gel Col. Chrom. Straight phase.

2) back-pressure regulation with] flash Chromatography

NPL (J. Chromatography?)

Please use (Vit D + Separation or isolation for Text Search

STAFF USE ONLY		Type of Search		Vendors and cost where applicable	
Searcher:	Ian	NA Sequence (#)		STN	✓
Searcher Phone #:	22504	AA Sequence (#)		Dialog	
Searcher Location		Structure (#)	✓	Questel/Orbit	
Date Searcher Picked Up	5/8	Bibliographic	✓	Dr.Link	
Date Completed:	5/8	Litigation		Lexis/Nexis	
Searcher Prep & Review Time:		Fulltext		Sequence Systems	
Clerical Prep Time:	15	Patent Family		WWW/Internet	
Online Time:	44	Other		Other (specify)	

PTO-1571.3 (6-03)

4

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=> fil reg
FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004
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Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES:     7 MAY 2004  HIGHEST RN 680859-76-1
DICTIONARY FILE UPDATES:    7 MAY 2004  HIGHEST RN 680859-76-1

TSCA INFORMATION NOW CURRENT THROUGH JANUARY 6, 2004

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

Crossover limits have been increased.  See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
http://www.cas.org/ONLINE/DBSS/registryss.html

=> d sta que l6
L4                STR
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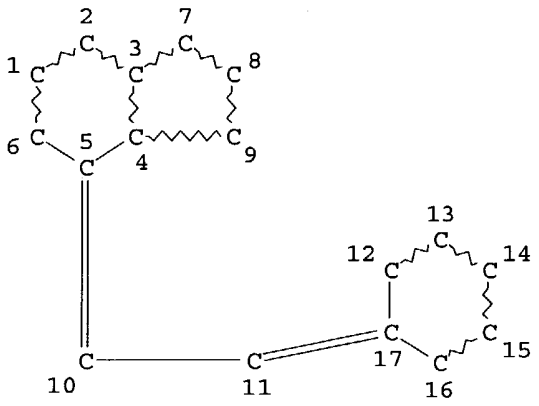


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NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS  17

STEREO ATTRIBUTES: NONE
L6          8414 SEA FILE=REGISTRY SSS FUL L4

100.0% PROCESSED  15155 ITERATIONS                      8414 ANSWERS
SEARCH TIME: 00.00.01


=> => d sta que l9
L7                STR
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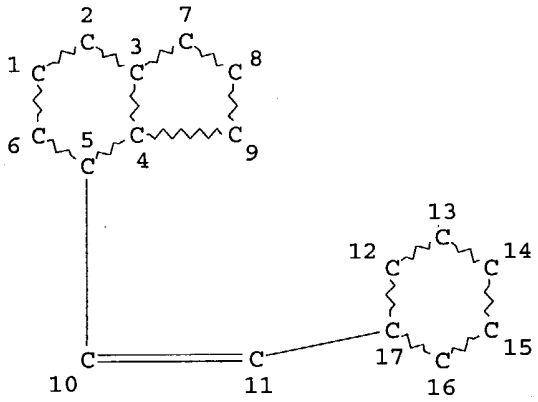


NODE ATTRIBUTES:
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 17

STEREO ATTRIBUTES: NONE
L9 662 SEA FILE=REGISTRY SSS FUL L7

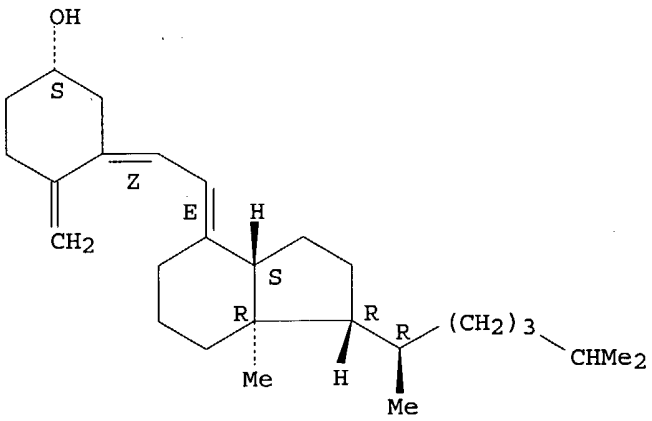
100.0% PROCESSED 203796 ITERATIONS 662 ANSWERS
SEARCH TIME: 00.00.02

=> d ide can l1

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
RN 67-97-0 REGISTRY
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Cholecalciferol (8CI)
OTHER NAMES:
CN 9,10-Secocholesta-5,7,10(19)-trien-3β-ol
CN Arachitol
CN Calciol
CN Colecalciferol
CN D3-Vigantol
CN Delsterol
CN Deparal
CN Devaron
CN FeraCol
CN Granuvit D3
CN NSC 375571
CN Oleovitamin D3
CN Quintox
CN Ricketon
CN Trivitan
CN Vi-De3
CN Videkhol
CN Vigorsan
CN **Vitamin D3**
CN Vitinc Dan-Dee-3
FS STEREOSEARCH

DR 8024-19-9, 8050-67-7
MF C27 H44 O
CI COM
LC STN Files: ADISNEWS, AGRICOLA, BEILSTEIN*, BIOBUSINESS, BIOSIS, BIOTECHNO, CABA, CANCERLIT, CAOLD, CAPLUS, CHEMCATS, CHEMLIST, CIN, DDFU, DRUGU, EMBASE, HODOC*, HSDB*, IFICDB, IFIPAT, IFIUDB, IPA, MRCK*, NIOSHTIC, PS, SPECINFO, TOXCENTER, USAN, USPAT2, USPATFULL, VETU
(*File contains numerically searchable property data)
Other Sources: EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)

Absolute stereochemistry.
Double bond geometry as shown.



PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

5260 REFERENCES IN FILE CA (1907 TO DATE)
492 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
5265 REFERENCES IN FILE CAPLUS (1907 TO DATE)
5 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:326651

REFERENCE 2: 140:320239

REFERENCE 3: 140:315647

REFERENCE 4: 140:309487

REFERENCE 5: 140:309381

REFERENCE 6: 140:309014

REFERENCE 7: 140:302745

REFERENCE 8: 140:302652

REFERENCE 9: 140:302466

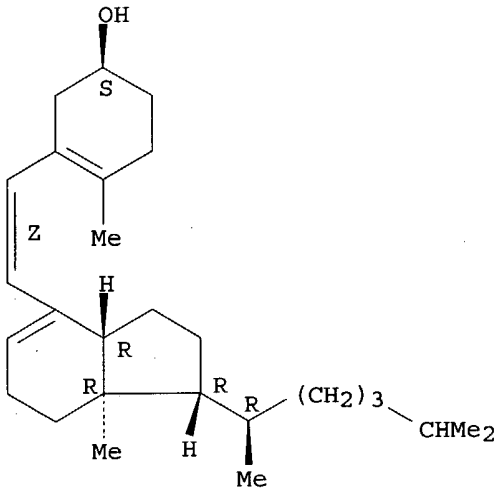
REFERENCE 10: 140:300743

=> d ide can l2

L2 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN

RN 1173-13-3 REGISTRY
CN 9,10-Secocholesta-5(10),6,8-trien-3-ol, (3β,6Z)- (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN 9,10-Secocholesta-5(10),6,8-trien-3β-ol (8CI)
CN Precalciferol (6CI)
CN **Previtamin D3 (7CI)**
OTHER NAMES:
CN Precalciferol3
CN Precholecalciferol
CN Previtamin D
FS STEREOSEARCH
MF C27 H44 O
LC STN Files: AGRICOLA, BEILSTEIN*, BIOBUSINESS, BIOSIS, CA, CAOLD, CAPLUS, CASREACT, CHEMINFORMRX, CHEMLIST, DDFU, DRUGU, EMBASE, IFICDB, IFIPAT, IFIUDB, MEDLINE, PROMT, PS, TOXCENTER, USPATFULL
(*File contains numerically searchable property data)
Other Sources: EINECS**
(**Enter CHEMLIST File for up-to-date regulatory information)

Absolute stereochemistry.
Double bond geometry as shown.



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233 REFERENCES IN FILE CA (1907 TO DATE)
9 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
234 REFERENCES IN FILE CAPLUS (1907 TO DATE)
12 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:301671

REFERENCE 2: 140:162982

REFERENCE 3: 140:16105

REFERENCE 4: 139:307389

REFERENCE 5: 138:255392

REFERENCE 6: 138:234042

REFERENCE 7: 138:136053

REFERENCE 8: 138:102954

REFERENCE 9: 137:302012

REFERENCE 10: 137:67030

=> d ide can l3

L3 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
RN 1406-16-2 REGISTRY
CN **Vitamin D (8CI, 9CI)** (CA INDEX NAME)
MF Unspecified
CI COM, MAN
LC STN Files: ADISNEWS, AGRICOLA, ANABSTR, BIOBUSINESS, BIOSIS, BIOTECHNO, CA, CABA, CANCERLIT, CAPLUS, CASREACT, CBNB, CEN, CHEMLIST, CIN, CSNB, DDFU, DIOGENES, DRUGU, EMBASE, IFICDB, IFIPAT, IFIUDB, IPA, MEDLINE, NAPRALERT, NIOSHTIC, PIRA, PROMT, RTECS*, TOXCENTER, USPAT2, USPATFULL, VETU
(*File contains numerically searchable property data)
Other Sources: EINECS**, NDSL**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
10574 REFERENCES IN FILE CA (1907 TO DATE)
833 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
10585 REFERENCES IN FILE CAPLUS (1907 TO DATE)

REFERENCE 1: 140:327125

REFERENCE 2: 140:327062

REFERENCE 3: 140:326651

REFERENCE 4: 140:320585

REFERENCE 5: 140:320583

REFERENCE 6: 140:320475

REFERENCE 7: 140:320433

REFERENCE 8: 140:320428

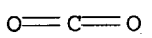
REFERENCE 9: 140:320350

REFERENCE 10: 140:320336

=> => d ide can l14

L14 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2004 ACS on STN
RN 124-38-9 REGISTRY
CN **Carbon dioxide (8CI, 9CI)** (CA INDEX NAME)
OTHER NAMES:
CN Carbon oxide (CO2)
CN Carbon-12 dioxide
CN Carbon-12C dioxide-16O2

CN Carbonic acid anhydride
CN Carbonic acid gas
CN Carbonic anhydride
CN Dry ice
CN Khladon 744
CN R 744
FS 3D CONCORD
DR 18923-20-1
MF C O2
CI COM
LC STN Files: ANABSTR, BIOSIS, CA, CAOLD, CASREACT, CHEMCATS, CHEMLIST, CIN, CSCHEM, CSNB, DIPPR*, GMELIN*, HODOC*, IFICDB, IFIPAT, IFIUDB, MEDLINE, PDLCOM*, RTECS*, SPECINFO, TOXCENTER, USAN, USPAT2, USPATFULL, VTB
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)



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176638 REFERENCES IN FILE CA (1907 TO DATE)
678 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
176812 REFERENCES IN FILE CAPLUS (1907 TO DATE)
21 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:331428

REFERENCE 2: 140:331427

REFERENCE 3: 140:330835

REFERENCE 4: 140:330508

REFERENCE 5: 140:330413

REFERENCE 6: 140:329684

REFERENCE 7: 140:329637

REFERENCE 8: 140:329394

REFERENCE 9: 140:329119

REFERENCE 10: 140:328988

=> d his

(FILE 'HOME' ENTERED AT 14:46:34 ON 08 MAY 2004)
SET COST OFF

FILE 'REGISTRY' ENTERED AT 14:46:51 ON 08 MAY 2004
E VITAMIN D3/CN
L1 1 S E3
E PREVITAMIN D3/CN
L2 1 S E3
E VITAMIN D/CN
L3 1 S E3

```
L4              STR
L5           50 S L4
L6         8414 S L4 FUL
                SAV TEMP L6 QAZI335/A
L7              STR L4
L8            0 S L7
L9          662 S L7 FUL
                SAV L9 TEMP QAZI335A/A
L10        9076 S L6,L9
L11        9074 S L10 NOT L1,L2
L12         366 S L11 AND ?VITAMIN?/CNS
L13        8708 S L11 NOT L12
L14           1 S CARBON DIOXIDE/CN

     FILE 'HCAPLUS' ENTERED AT 14:52:16 ON 08 MAY 2004
L15        5267 S L1
L16         235 S L2
L17       12688 S (VIT OR VITAMIN)(L)D3
L18         136 S (VIT OR VITAMIN)(L)D 3
L19        2247 S CHOLECALCIFEROL#
L20          14 S COLECALCIFEROL#
L21         249 S (PREVIT OR PREVITAMIN OR PRE()(VIT OR VITAMIN))(L)D3
L22           6 S (PREVIT OR PREVITAMIN OR PRE()(VIT OR VITAMIN))(L)D 3
L23          83 S PRECALCIFEROL# OR PRE CALCIFEROL#
L24         125 S PREVITAMIN D
L25       10602 S L3
L26       29557 S VITAMIN D#
L27       16535 S L12
L28        3123 S L13
L29       36305 S L15-L28
                E JOHANNSEN M/AU
L30          17 S E3,E8
L31           2 S L30 AND L29
L32         270 S L29 AND (LAROCHE? OR LA ROCHE? OR HOFFMANN?)/PA,CS
L33           1 S US20010001801/PN OR EP98-111490/AP,PRN
L34      176984 S L14
L35      441586 S CARBON DIOXIDE OR CO2
L36         204 S CARBONDIOXIDE
L37         184 S L29 AND L34-L36
L38           4 S L37 AND L31,L32,L33
                E CHROMATOGRAPHY/CT
L39           1 S L37 AND E30
L40           3 S L37 AND E110
L41           2 S L37 AND E145,E151,E154
L42           0 S L37 AND E158
L43           2 S L37 AND E27-E29
                E E3+ALL
L44           2 S L37 AND E4-E6
L45          12 S L37 AND E3+NT
                E SILICA GEL/CT
L46           2 S L37 AND E4-E28
                E E3+ALL
L47           2 S L37 AND E16,E15+NT
L48          18 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT?)
L49           1 S L37 AND FLASH?
L50          23 S L33,L38-L49
                E SEPARATION/CT
L51         561 S E3+OLD,NT,PFT AND L29
                E PURIFICATION/CT
L52           9 S E3+OLD,NT,PFT AND L29
                E ISOLATION/CT
L53         561 S L51-L52
L54          19 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
```

```
L55           0 S L37 AND (BACKPRESSUR? OR BACK PRESSUR?)
L56          22 S L37 AND ?CHROMATOG?
L57          30 S L54,L56,L50
L58          20 S L57 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L59         473 S (PROVIT? OR PRO VIT?)()D#
L60         172 S L59 NOT L29
L61         163 S L60 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L62          23 S L61 AND ?CHROMATO?
L63           0 S L61 AND (BACKPRESSUR? OR BACK PRESSUR?)
L64           0 S L61 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
L65           7 S L61 AND L34-L36
L66           2 S L61 AND CHROMATOGRAPHY+OLD,NT,PFT/CT
L67           2 S L61 AND SEPARATION+OLD,NT,PFT/CT
L68           0 S L61 AND PURIFICATION+OLD,NT,PFT/CT
L69          49 S L62,L65-L67,L58
L70          49 S L69 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L71          10 S L57 NOT L70
L72           8 S L70 AND ?CRITIC?
L73          27 S L70 AND L34-L36
L74          27 S L72,L73
L75          22 S L70 NOT L74
                SEL DN AN L74 25
L76          26 S L74 NOT E1-E3
L77          36 S L71,L76
L78          12 S L77 AND (?RADIAT? OR UV OR ULTRAVIOL? OR ULTRA VIOL?)
L79          36 S L77,L78

     FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004

=> fil hcaplus
FILE 'HCAPLUS' ENTERED AT 15:18:23 ON 08 MAY 2004
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FILE COVERS 1907 - 8 May 2004  VOL 140 ISS 20
FILE LAST UPDATED: 7 May 2004  (20040507/ED)

 This file contains CAS Registry Numbers for easy and accurate
 substance identification.


=> d l79 all hitstr tot

L79  ANSWER 1 OF 36  HCAPLUS  COPYRIGHT 2004 ACS on STN
AN   2003:855901  HCAPLUS
DN   139:354553
ED   Entered STN:  31 Oct 2003
TI   Extraction of lipophilic compounds for cosmetic and pharmaceutical uses
IN   Catchpole, Owen John; MacKenzie, Andrew Douglas; Grey, John Bertram
PA   Industrial Research Limited, N. Z.
SO   PCT Int. Appl., 49 pp.
```

CODEN: PIXXD2
DT Patent
LA English
IC ICM C07C067-56
ICS C11B007-00; C11C001-08
CC 63-8 (Pharmaceuticals)
Section cross-reference(s): 9, 17, 62
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2003089399	A1	20031030	WO 2003-NZ62	20030411

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRAI NZ 2002-518504 A 20020422
AB A process for extracting a wide range of lipophilic compds. from urea-containing solns. is described. The process utilizes a near-critical fluid as the extraction solvent. The process is particularly applicable to the extraction of polyunsatd. fatty acids from the filtrate obtained upon urea fractionation, as employed in the processing of fish and other oils. In contrast to known processes, the lipophilic compds. may be extracted without the use of non-food grade solvents, and are suitable for pharmaceutical and cosmetic uses. Thus, free fatty acids (FFA) were recovered from fish oil glycerides followed by urea complexation. The polyunsatd. fatty acids content was 40.8%. The total recovery of FFA was 86%.
ST extn lipophilic compd cosmetic pharmaceutical
IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(Biota orientalis; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(Currant (Ribes nigrum); extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT Alcohols, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(C1-4; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(Echium; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);

PROC (Process); USES (Uses)
(Limnanthes alba seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(Lunarium; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(animal, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(borage seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(esters, with C1-4 alcs.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(evening primrose; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Extraction
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Carotenes, biological studies
Fatty acids, biological studies
Glycerides, biological studies
Hydrocarbons, biological studies
Sterols
Vitamins
Waxes
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Vitamins
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(fat-soluble; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Alcohols, biological studies
Amides, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(fatty; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study);

PROC (Process); USES (Uses)
(fish, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Hydrocarbons, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(fluoro; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Lipids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(monounsatd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(polyunsatd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(pumpkin seed; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(saturated; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(saw palmetto; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fatty acids, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(unsatd.; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT Fats and Glyceridic oils, biological studies
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(vegetable, hydrolyzates; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)

IT 56-81-5D, Glycerol, ethers 60-33-3, 9,12-Octadecadienoic acid (9Z,12Z)-, biological studies 64-17-5, Ethanol, biological studies 111-02-4, Squalene 112-80-1, Oleic acid, biological studies 115-10-6, Dimethyl ether 463-40-1 472-61-7, Astaxanthin 502-65-8, Lycopene 506-26-3 **1406-16-2, Vitamin D** 1406-18-4, Vitamin E 6217-54-5 7235-40-7, β-Carotene 10417-94-4 11103-57-4, Vitamin A

RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT 57-13-6, Urea, processes 74-84-0, Ethane, processes 74-85-1, Ethylene, processes 74-98-6, Propane, processes 106-97-8, Butane, processes 115-07-1, Propylene, processes 811-97-2, 1,1,1,2,-Tetrafluoroethane 2551-62-4, Sulfur hexafluoride 10024-97-2, Nitrous oxide, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
IT **124-38-9, Carbon dioxide**, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(**supercrit.**; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
RE.CNT 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Babcock-Hitachi Kk; JP 62-072793 A 1985
(2) Jgc Corp; JP 60-214757 A 1985 HCAPLUS
(3) Nilsson, W; J Am Oil Chem Soc 1988, V65(1), P109 HCAPLUS
(4) Norsk Hydro Asa; WO 0110809 A 2001 HCAPLUS
(5) Shahidi, F; Trends in Food Science & Technology 1998, V9, P230 HCAPLUS
IT **1406-16-2, Vitamin D**
RL: COS (Cosmetic use); PEP (Physical, engineering or chemical process); PYP (Physical process); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
IT **124-38-9, Carbon dioxide**, processes
RL: PEP (Physical, engineering or chemical process); PYP (Physical process); PROC (Process)
(**supercrit.**; extraction of lipophilic compds. for cosmetic and pharmaceutical uses)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

L79 ANSWER 2 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2003:845155 HCAPLUS
DN 140:82892
ED Entered STN: 29 Oct 2003
TI Calculation of Solid Solubility of Complex Molecules in **Supercritical Carbon Dioxide** using a Solution Model Approach
AU Cheng, Jaw-Shin; Tang, Muoi; Chen, Yan-Ping
CS Department of Chemical Engineering, National Taiwan University, Taipei, Taiwan
SO Molecular Simulation (2003), 29(12), 749-754
CODEN: MOSIEA; ISSN: 0892-7022
PB Taylor & Francis Ltd.
DT Journal
LA English
CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 65, 69
AB Solid solubility of complex mols. in **supercrit. carbon**

dioxide was calculated using a solution model approach. These solutes include the biol. compds. of antioxidants, steroids, vitamins, and quinones. The modified AD (m-AD) activity coefficient model proposed by Cheng, J.S. et al. (2002) coupled with the Flory-Huggins equation was employed. The molar volume of the solutes in the **supercrit.** phase and the solid-fluid interaction parameter were optimally fitted for each solid component. Satisfactory results were obtained from the m-AD model with three or four parameters. The overall accuracy in this study is comparably good to that from the conventional equation of state method or the semi-empirical correlation equation. This result suggests a feasible high-pressure solid solubility calcn. with the application of a theor. based solution model.

ST org compd solid soly **supercrit carbon dioxide** soln modeling

IT Activity (thermodynamic)
Antioxidants
Molar volume
Simulation and Modeling, physicochemical
Solid solubility
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

IT Quinones
Steroids, properties
Vitamins
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

IT Solvents
(**supercrit.**; calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

IT **50-14-6**, **Vitamin D2** 50-29-3, DDT, properties
50-81-7, Ascorbic acid, properties 57-83-0, Progesterone, properties
57-88-5, Cholesterol, properties 58-22-0, Testosterone **67-97-0**, **Vitamin D3** 71-58-9, Medroxyprogesterone acetate
83-48-7, Stigmasterol 84-65-1, 9,10-Anthraquinone 94-75-7, 2,4-Dichlorophenoxy acetic acid, properties 106-51-4, 1,4-Benzoquinone, properties 118-74-1, Hexachlorobenzene 121-79-9, Propyl gallate
124-38-9, **Carbon dioxide**, properties
130-15-4, 1,4-Naphthoquinone 137-66-6, Ascorbyl palmitate 525-82-6, Flavone 577-85-5, 3-Hydroxyflavone 1166-52-5, Dodecyl gallate
11103-57-4, Vitamin A 22204-53-1, Naproxen
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

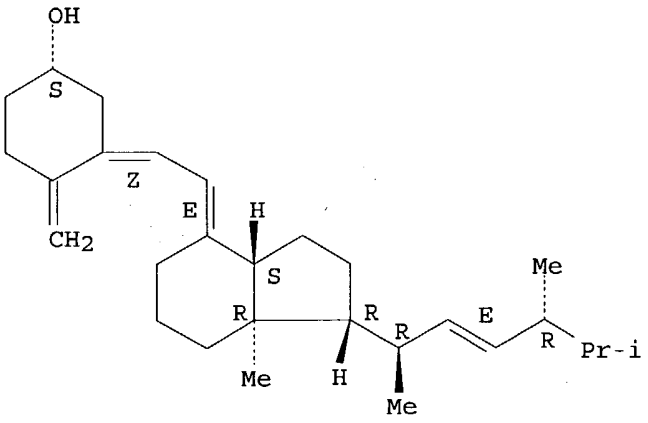
(1) Alessi, P; Ind Eng Chem Res 1996, V35, P4718 HCAPLUS
(2) Aranovich, G; J Chem Phys 1996, V105, P7059 HCAPLUS
(3) Ashour, I; Fluid Phase Equilib 2000, V167, P41 HCAPLUS
(4) Bush, D; Fluid Phase Equilib 1998, V150-151, P479
(5) Cheng, J; Fluid Phase Equilib 2002, V194-197, P483 HCAPLUS
(6) Cheng, J; Fluid Phase Equilib, In final revision 2003
(7) Chrastil, J; J Chem Phys 1982, V86, P3016 HCAPLUS
(8) Cortesi, A; J Supercrit Fluids 1999, V14, P139 HCAPLUS
(9) Coutsikos, P; J Chem Eng Data 1997, V42, P463 HCAPLUS
(10) Ely, J; J Chem Thermodyn 1989, V21, P879 HCAPLUS
(11) Iwai, Y; J Chem Eng Jpn 1992, V25, P757 HCAPLUS
(12) Jiang, C; J Supercrit Fluids 1998, V12, P1 HCAPLUS
(13) Johannsen, M; J Chem Eng Data 1997, V42, P106 HCAPLUS
(14) Kosal, E; J Supercrit Fluids 1992, V5, P169 HCAPLUS

(15) Macnaughton, S; Ind Eng Chem Res 1994, V33, P2757 HCAPLUS
(16) Madras, G; J Chem Eng Data 1993, V38, P422 HCAPLUS
(17) Mendez-Santiago, J; Fluid Phase Equilib 1999, V158-160, P501 HCAPLUS
(18) Schmitt, W; J Chem Eng Data 1986, V31, P204 HCAPLUS
(19) Ting, S; Ind Eng Chem Res 1993, V32, P1471 HCAPLUS
(20) Uchiyama, H; J Chem Eng Data 1997, V42, P570 HCAPLUS
(21) Wong, J; Biotechnol Prog 1986, V2, P29 HCAPLUS
(22) Yalkowsky, S; Ind Eng Chem Fundam 1979, V18, P108 HCAPLUS
(23) Yun, S; Ind Eng Chem Res 1991, V30, P2476 HCAPLUS

IT **50-14-6**, **Vitamin D2 67-97-0**, **Vitamin D3 124-38-9**, **Carbon dioxide**, properties
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PYP (Physical process); PROC (Process)
(calcn. of organic compound solid solubility in **supercrit. carbon dioxide** using solution model approach)

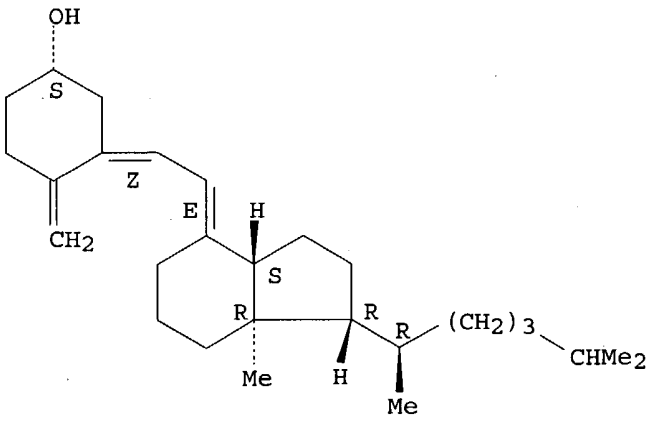
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.

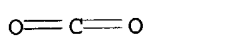


RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 3 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2003:651925 HCAPLUS
DN 139:306776
ED Entered STN: 21 Aug 2003
TI Fat-soluble vitamin extraction by analytical **supercritical carbon dioxide**
AU Perretti, Giuseppe; Marconi, Ombretta; Montanari, Luigi; Fantozzi, Paolo
CS Department of Food Science, University of Perugia, Perugia, I-06126, Italy
SO Journal of the American Oil Chemists' Society (2003), 80(7), 629-633
CODEN: JAOCA7; ISSN: 0003-021X
PB AOCS Press
DT Journal
LA English
CC 17-1 (Food and Feed Chemistry)
AB Extraction of fat-soluble vitamins (A, D, E, and β-carotene) by **supercrit. carbon dioxide** (SC-**CO2**) was tested to replace conventional liquid extraction methods, which require large vols. of organic solvents. **Supercrit.** fluid extraction (SFE) is a rapid extraction technique for fat-soluble vitamins enabling them to be accurately determined using only small vols. of organic solvents. Extns. were performed on ultra-high-temperature sterilized milk, milk powder, pork, liver pate (pate de fois), infant formula, and canned baby food to compare the methods. The proposed method is based on the extraction of fat-soluble vitamins and their esters by using SC-**CO2** with methanol as a modifier. HPLC anal. using photometric detection was used for the vitamin anal. The results showed no significant differences between extraction methods. The proposed SFE method appears to be useful as a substitute for the traditional organic solvent method, mainly for vitamin A and γ-tocopherol.
ST food analysis **supercrit** extn fat soluble vitamin
IT Dairy products
Food analysis
Meat
(fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Food
(infant; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT Extraction
(**supercrit.**; fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
IT 59-02-9, α-Tocopherol **67-97-0, Vitamin D3** 7235-40-7, β-Carotene 7616-22-0, γ-Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble **vitamin** extraction by anal. **supercrit. carbon dioxide**)
IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(fat-soluble vitamin extraction by anal. **supercrit. carbon**

dioxide)

RE.CNT 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

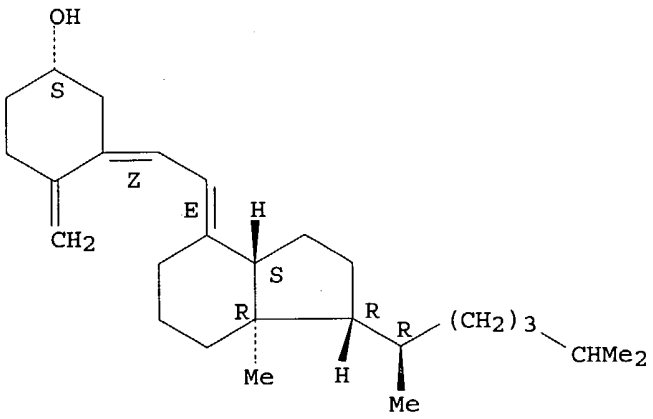
(1) AOAC; Official Methods of Analysis, 16th edn 1996, sec 45.1.02
(2) AOAC; Official Methods of Analysis, 16th edn 1996, sec 45.1.26
(3) AOAC; Official Methods of Analysis, 16th edn 1996, sec 45.1.21
(4) AOAC; Official Methods of Analysis, 16th edn 1996, sec 45.1.03
(5) Anon; Official Methods and Recommended Practices of the American Oil Chemists' Society 1996, Method Am 2-93
(6) Ball, G; Fat-Soluble Vitamin Assays in Food Analysis: A Comprehensive Review 1988, P64
(7) Berg, H; J Biochem Biophys Methods 2000, V43, P391 HCAPLUS
(8) Borsje, B; J Assoc Off Anal Chem 1982, V65, P1225 HCAPLUS
(9) Cohen, H; J Agric Food Chem 1978, V26, P1210 HCAPLUS
(10) De Vries, E; J Assoc Off Anal Chem 1982, V65, P1228 HCAPLUS
(11) Eller, F; Semin Food Anal 1996, V1(2), P145 HCAPLUS
(12) Gordon, M; Encyclopedia of Food Science, Food Technology and Nutrition 1993, P212
(13) Han, Y; Chin Chem Lett 1991, V2, P649 HCAPLUS
(14) King, J; J Agric Food Chem 1996, V44, P2700 HCAPLUS
(15) King, J; J Food Sci 1993, V58, P950 HCAPLUS
(16) Mathiasson, L; Development and Intercomparison of Improved Methods for Determination of Fat Soluble Vitamins in Food by Replacement of Hazardous Organic Solvents with Supercritical Carbon Dioxide. Final report EU 2000, Project code SMT4-CT96-2089
(17) Mathiasson, L; Food Addit Contam 2002, V19, P632 HCAPLUS
(18) Perretti, G; Lebensm Wiss Technol (in press)
(19) Schneiderman, M; J Assoc Off Anal Chem 1988, V71, P815 HCAPLUS
(20) Stancher, B; Ind Aliment 1997, V36, P621 HCAPLUS
(21) Taylor, L; Supercritical Fluid Extraction 1996, P124
(22) The Royal Society of Chemistry and Ministry of Agriculture Fisheries and Food; McCance and Widdowson's The Composition of Foods 1991, P172
(23) Turner, C; J Chromatogr 2000, V874, P275 HCAPLUS

IT **67-97-0, Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble **vitamin** extraction by anal. **supercrit. carbon dioxide**)

RN 67-97-0 HCAPLUS

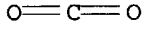
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)

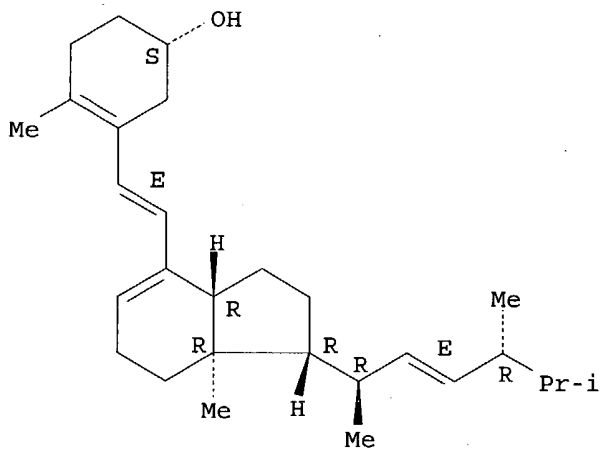
(fat-soluble vitamin extraction by anal. **supercrit. carbon dioxide**)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 4 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2003:462432 HCAPLUS
DN 139:58078
ED Entered STN: 17 Jun 2003
TI Preparative **supercritical** fluid **chromatography** as an example of vitamin separation
AU Buss, Vessela
CS Aschaffenburg, Germany
SO Fortschritt-Berichte VDI, Reihe 3: Verfahrenstechnik (2003), 778, i-xiii,1-138
CODEN: FVVEFK; ISSN: 0178-9503
PB VDI Verlag GmbH
DT Journal
LA German
CC 64-2 (Pharmaceutical Analysis)
AB The use of **supercrit.** fluid **chromatog.** (SFC) in packed colums was investigated. Zorbax Pro 10-60 CN was applied as stationary phase to sep. in anal. scale several mixts. containing α-tocopherol, the **vitamins D2** and **D3**, or 7-dehydrocholesterol. Pressure and composition of the mobile phase were optimized. A mixture of 20% α-tocopherol and 80% **vitamin D3** was separated by preparative **chromatog.** The separation was carried out with **CO2** and 0.12% (weight) EtOH as modifier at 13.8 MPa and 313.15 K. Purities of >99% **vitamin D3** and 60% α-tocopherol were obtained. Adsorption isothermes of the components were determined by the peak maxima method to predict elution profiles. The construction of a SFC apparatus was described for pressures up to 25 MPa and temps. up to 368.15 K.
ST **supercrit** fluid **chromatog** tocopherol **vitamin D**
IT Adsorption
(isotherm; preparative **supercrit.** fluid **chromatog.** as an example of vitamin separation)
IT Preparative **chromatography**
Simulation and Modeling, physicochemical
Supercritical fluid chromatography
(preparative **supercrit.** fluid **chromatog.** as an example of vitamin separation)
IT **50-14-6, Vitamin D2 67-97-0, Vitamin D3** 434-16-2, 7-Dehydrocholesterol 10191-41-0, D,L-α-Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(preparative **supercrit.** fluid **chromatog.** as an example of **vitamin** separation)
RE.CNT 132 THERE ARE 132 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Aaltonen, O; Pharmaceutical Tech Europe 1998, V8, PXLII
(2) Anon; IUPAC Recommendations 1997
(3) Anon; International Thermodynamic Tables of the Fluid State, Carbon Dioxide 1976, P3
(4) Anon; Synthesis Using Supercritical Fluids 1999
(5) Bartle, K; J Chromatogr A 1995, V697, P579 HCAPLUS
(6) Bartle, K; Supercritical Fluid Chromatography 1988
(7) Belitz, H; Lehrbuch der Lebensmittelchemie 1987

(8) Bellot, J; Process Biochemistry 1991, V26, P363 HCAPLUS
(9) Berger, C; J Chromatogr 1990, V505(1), P37 HCAPLUS
(10) Berger, T; 5th International Symposium on Supercritical Fluids 2000
(11) Berger, T; Packed column SFC 1995
(12) Bevan, C; Process Scale Liquid Chromatography 1995
(13) Blilie, A; Anal Chem 1985, V57, P2239 HCAPLUS
(14) Blum, A; Chirality 1994, V6, P302 HCAPLUS
(15) Bridgeman, P; Phys Rev 1914, V3(126), P153
(16) Bridgeman, P; Phys Rev 1934, V46, P930
(17) Bridgeman, P; The Physics of High Pressure 1952
(18) Broughton, D; Ind Eng Chem 1948, V40, P1506 HCAPLUS
(19) Brunner, G; Gas Extraction 1994
(20) Brunner, G; Sep Sci Tech 1982, V17(1), P199 HCAPLUS
(21) Clark, A; Theory of adsorption 1970
(22) Coleman, K; Isolation & Purification 1999, V3, P9 HCAPLUS
(23) Colin, H; Preparative and Production Scale Chromatography 1993
(24) Cremer, E; Angew Chemie 1961, V73, P461 HCAPLUS
(25) Czok, M; Comp Chem Eng 1990, V14(2), P1435
(26) Dean, T; Chromsymp 1984, P1544
(27) Deemter van, J; Chem Eng Sci 1956, V5, P271
(28) Denisova, L; J Analytical Chemistry 1997, V52(9), P877 HCAPLUS
(29) Depta, A; Dissertation, Technische Universitat Hamburg-Harburg 2000
(30) Doguet, L; 2eme Colloque sur Les Fluides Supercritiques 1991, P219
(31) Doguet, L; Proceedings of the 2nd International Symposium on Supercritical Fluids 1991
(32) Ettre, L; Pure & Appl Chem 1993, V65(4), P819 HCAPLUS
(33) Filimonov, V; Russian J of Physical Chemistry 1994, V68(10), P1665
(34) Finlayson, B; Nonlinear analysis in chemical engineering 1980
(35) Fuchs, G; J Chromatogr 1992, V623, P329 HCAPLUS
(36) Gagniard de la Tour, C; Ann Chem Phys Ser 1822, V2(21), P127
(37) Gagniard de la Tour, C; Ann Chem Phys Ser 1822, V22, P410
(38) Geiser, F; J Chromatogr A 1999, V865, P227 HCAPLUS
(39) Gervais, D; J Chromatogr A 1995, V708, P41 HCAPLUS
(40) Giddings, J; Science 1968, V162, P67 HCAPLUS
(41) Giese, T; Dissertation, Technische Universitat Hamburg-Harburg 2002
(42) Glueckauf, E; J Chem Soc 1947, P1302
(43) Golshan-Shirazi, S; J Chromatogr A 1994, V658, P149 HCAPLUS
(44) Golshan-Shirazi, S; Theoretical Advancement in Chromatography and Related Separation Techniques 1992
(45) Golshan-Shirazi, S; Theoretical Advancement in Chromatography and Related Separation Techniques 1992
(46) Gomis, D; Anal Chem Asta 1995, V315, P177
(47) Gu, T; Mathematical Modeling and Scale-up of Liquid Chromatography 1995
(48) Guan, H; J Chromatogr A 1994, V659, P27 HCAPLUS
(49) Guan-Sajonz, H; J Chromatogr A 1996, V743, P247 HCAPLUS
(50) Guiochon, G; Fundamentals of Preparative and Nonlinear Chromatography 1994
(51) Guiochon, G; J Chromatogr 1996, V734, P423 HCAPLUS
(52) Helfferich, F; Multicomponent chromatography 1970
(53) Heuer, C; Dissertation, Technische Universitat Berlin 1996
(54) Hewlett Packard Company; Hewlett-Packard: User Guide for HP G1205A SFC 1994
(55) Higashidate, S; J Chromatogr 1990, V515, P295 HCAPLUS
(56) Hildebrand, J; The Solubility of Nonelectrolytes 1950
(57) Hill, T; An introduction to statistical thermodynamics 1986
(58) Ibanez, E; J Agric Food Chem 1995, V43, P2667 HCAPLUS
(59) Janssen, J; HRC 1989, V12, P645 HCAPLUS
(60) Jentoft, R; Anal Chem 1972, V44(4), P681 HCAPLUS
(61) Johannsen, M; EP 969001 A2 2000 HCAPLUS
(62) Johannsen, M; Dissertation, Technische Universitat Hamburg-Harburg 1995
(63) Jonsson, J; J Chromatogr 1987, V408(1), P1
(64) Josic, D; Ind Eng Chem Res 1999, V38, P333 HCAPLUS
(65) Jusforgues, P; Proceedings of SPICA 98 1998
(66) Jusforgues, P; Process Scale Liquid Chromatography 1995

(67) Jusforgues, P; Supercritical Fluid Chromatography with Packed Columns: Techniques and Applications 1998
(68) Kaczmarski, K; Computers Chem Eng 1995, V20(1), P49
(69) Kast, W; Adsorption aus der Gasphase 1988
(70) Katti, A; Advances in Chromatography 1992, V31, P1 HCAPLUS
(71) Klatt, K; Chemic Ingenieur Technik 1999, V71, P555 HCAPLUS
(72) Klawiter, J; J Chromatogr 1982, V243, P207 HCAPLUS
(73) Klesper, E; J Org Chem 1962, V27, P700 HCAPLUS
(74) Kniep, H; Dissertation, Otto-von-Guericke-Universitat Magdeburg 1998
(75) Knox, J; J Chromatogr 1986, V363, P1 HCAPLUS
(76) Koh, J; J Chromatogr A 1998, V813, P223 HCAPLUS
(77) Kot, A; J Chromatogr 1994, V32(10), P439 HCAPLUS
(78) LeVeque, R; Numerical methods for conservative laws 1990
(79) Lembke, P; Supercritical Fluid Chromatography with Packed Columns: Techniques and Applications 1998
(80) Leutert, T; J Chromatogr 1984, V292, P333 HCAPLUS
(81) Leyendecker, D; Supercritical Fluid Chromatography 1988
(82) Lin, B; Sep Sci Techn 1989, V24, P809 HCAPLUS
(83) Linder, H; J Chromatogr Sci 1976, V14, P234 HCAPLUS
(84) Lubbert, M; 2nd International Meeting of HPCE 2001
(85) Manninen, P; JAOCS 1995, V72(9), P1001 HCAPLUS
(86) Matsumoto, K; Chromatographia 1986, V21, P617 HCAPLUS
(87) Meyer, V; Praxis der Hochleistungs-Flussigchromatographie 1992
(88) Miyabe, K; J Chromatogr A 2000, V872, P1 HCAPLUS
(89) Mothes, H; Chem Eng Proc 1984, V18, P323 HCAPLUS
(90) Moutaftchieva, V; Interner Bericht 1997
(91) Myers, A; AIChE 1965, V11, P121 HCAPLUS
(92) Neumuller, O; Rompps Chemie-Lexikon 1979
(93) Newburger, J; J Chromatogr 1990, V523, P63 HCAPLUS
(94) Perrut, M; FR 8209649 1982
(95) Perrut, M; J Chromatogr A 1994, V658, P293 HCAPLUS
(96) Pettinello, G; 5th Conference on Supercritical Fluids and their Applications 1999
(97) Pietzonka, W; Dissertation, ETH-Zurich 1997
(98) Qian, H; J Chrmatogr A 1998, V825, P127 HCAPLUS
(99) Radke, C; AIChE 1972, V18, P761 HCAPLUS
(100) Reichmann, F; Dissertation, Technische Universitat Hamburg-Harburg 2001
(101) Reid, R; The Properties of Gases and Liquids 1987
(102) Rhee, H; First order partial differential equations 1989
(103) Rizzolo, A; J Chromatogr 1992, V624, P103 HCAPLUS
(104) Rouchon, P; Sep Sci Techn 1987, V22(8-10), P1793
(105) Rouquerol, J; Pure & Appl Chem 1994, V66(8), P1739 HCAPLUS
(106) Ruthven, D; Principles of Adsorption and Adsorption Processes 1984
(107) Saito, M; J Chromatogr 1990, V505, P257 HCAPLUS
(108) Sarker, M; J Chromatogr A 1996, V741, P165 HCAPLUS
(109) Sarker, M; J Chromatogr A 1996, V719, P275 HCAPLUS
(110) Schaffner, D; Dissertation, ETH Zurich 1993
(111) Schoenmakers, P; Supercritical Fluid Chromatography 1988
(112) Scholsky, K; J Chem Ed 1989, V66, P989 HCAPLUS
(113) Schulte, M; SilicaRods as novel non-particular stationary phases for preparative batch- and SMB-chromatography 1998, VFOA6, P365
(114) SeideI-Morgenstem, A; Mathematische Modellierung der praprativen Flussigkeitschromatographie 1995
(115) Seidel-Morgenstern, A; Proceedings of the European Meeting on the Simulated Moving Bed Process 1993
(116) Shi, T; Proceedings of the 5th International Symposium on Supercritical Fluids 2000
(117) Sie, S; Sep Sci 1967, V2, P729 HCAPLUS
(118) Sing, K; Pure & Appl Chem 1985, V57(4), P603 HCAPLUS
(119) Stanley, B; J Chromatogr A 1996, V741, P175 HCAPLUS
(120) Stanley, B; J Chromatogr A 1997, V761, P41 HCAPLUS
(121) Storck, M; Dissertation, RWTH Aachen 1994
(122) Terfloth, G; J Chromatogr A 2001, V906, P301 HCAPLUS



L79 ANSWER 12 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1998:253034 HCAPLUS
DN 128:312926
ED Entered STN: 04 May 1998
TI Process for the manufacture of a powdery preparation
IN Bausch, Alexander; Peter, Siegfried K. F.; Steiner, Kurt; Stoller, Hans Jorg; Weidner, Eckhard
PA F. **Hoffmann-La Roche** A.-G., Switz.
SO PCT Int. Appl., 27 pp.
CODEN: PIXXD2
DT Patent
LA English
IC A61K-914; A61K009-72; A61K310-15
CC 63-6 (Pharmaceuticals)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
PI	WO 9816204	A1	19980423	WO 1997-EP5621	19971013	<--
	W: BR, CN, ID, JP					
	RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE					
	CN 1233169	A	19991027	CN 1997-198781	19971013	<--
	EP 952820	A1	19991103	EP 1997-945817	19971013	<--
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL					
	BR 9713258	A	20000328	BR 1997-13258	19971013	<--
	JP 2001505190	T2	20010417	JP 1998-518006	19971013	<--
	EP 1097705	A2	20010509	EP 2000-121676	19971013	<--
	R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL					
	JP 2001226262	A2	20010821	JP 2000-312886	19971013	<--
	CN 1394597	A	20030205	CN 2000-131456	20001017	<--
	US 2001000143	A1	20010405	US 2000-726876	20001130	<--
	US 2001031282	A1	20011018	US 2000-726875	20001130	<--
	US 2002086059	A1	20020704	US 2001-45270	20011023	<--
PRAI	EP 1996-116420	A	19961014	<--		
	CH 1996-2895	A	19961125	<--		
	US 1997-953952	B1	19971010	<--		
	EP 1997-945817	A3	19971013	<--		
	JP 1998-518006	A3	19971013	<--		
	WO 1997-EP5621	W	19971013	<--		
	US 2000-726876	B1	20001130			

AB The invention concerns a process for the manufacture of powdery active substance selected from the groups of pharmaceuticals, pharmaceutical precursors, diagnostics, fine chems. or carotenoids, especially β-carotene, characterized by (1) dissolving the active substance in di-Me ether under

elevated pressure and temperature conditions, (2) **flash**-decompressing the thus-formed solution in an expansion apparatus, and (3) separating the powdery solid particles formed in the expansion from the di-Me ether liberated. Also described is a process for the manufacture of a powdery preparation containing the active ingredients distributed in a matrix component consisting of at least one adjuvant. Trans-β-carotene 167 g were stirred into 500 g of liquid polyethylene glycol at 90° and the mixture and 350 g di-Me ether were charged into an autoclave. The solution was passed through the inner nozzle and the gas liberated in the spray tower was fed to a cyclone together with the solids. After completion of the spray procedure, the spray tower was opened. Finely divided coppt. of polyethylene glycol and β-carotene were taken off.

ST carotene dimethyl ether adjuvant pulverization

IT Drug delivery systems
(powders; process for manufacture of powdery preparation to improve bioavailability and color intensity)

IT Gums and Mucilages
Hydrocolloids
(process for manufacture of powdery preparation to improve bioavailability and color intensity)

IT Carotenes, biological studies
Fats and Glyceridic oils, biological studies
Gelatins, biological studies
Polymers, biological studies
Polyoxyalkylenes, biological studies
Polysaccharides, biological studies
Proteins, general, biological studies
Vitamins
Waxes
RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)
(process for manufacture of powdery preparation to improve bioavailability and color intensity)

IT 79-10-7D, Acrylic acid, esters, polymers 115-10-6, Dimethyl ether **124-38-9**, **Carbon dioxide**, biological studies 439-14-5, Diazepam 723-46-6, Sulfamethoxazole 738-70-5, Trimethoprim 1812-30-2, Bromazepam 4759-48-2, Isotretinoin 7235-40-7, β-Carotene 7481-89-2, Zalcitabine 9003-39-8, PVP 22204-53-1, Naproxen 25322-68-3, Polyethylene glycol **32222-06-3**, Calcitriol 53230-10-7, Mefloquine 59467-70-8, Midazolam 59804-37-4, Tenoxicam 71320-77-9, Moclobemide 73384-59-5, Ceftriaxone 74103-06-3, Ketorolac 82410-32-0, Ganciclovir 88768-40-5, Cilazapril 96829-58-2, Orlistat 127779-20-8, Saquinavir 128794-94-5, Mycophenolate mofetil 134308-13-7, Tolcapone 144412-49-7, Lamifiban 147536-97-8, Bosentan 159989-65-8, Nelfinavir mesylate
RL: THU (Therapeutic use); BIOL (Biological study); USES (Uses)
(process for manufacture of powdery preparation to improve bioavailability and color intensity)

RE.CNT 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Basf Ag; DE 2943267 A1 1981 HCAPLUS
(2) Bayer Ag; CH 667203 A5 1988 HCAPLUS
(3) Chang, C; Aiche Journal 1989, V35(11), P1876 HCAPLUS
(4) Jay, A; The Journal of Supercritical Fluids 1992, V5(4), P274 HCAPLUS
(5) Marigen S A; WO 9518605 A1 1995 HCAPLUS
(6) Peter, S; WO 9615133 A1 1996 HCAPLUS
(7) Weidner, E; WO 9521688 A1 1995 HCAPLUS

IT **124-38-9**, **Carbon dioxide**, biological studies
32222-06-3, Calcitriol

with a saturator column. With this new mixing device, we could get good resolution for vitamins which are difficult to sep. with pure **CO2.**

ST vitamin **supercrit** fluid **chromatog**

IT **Supercritical fluid chromatography**
(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT 50-81-7, Ascorbic acid, analysis 59-43-8, Vitamin B1, analysis
59-67-6, Nicotinic acid, analysis 83-88-5, Vitamin B2, analysis
98-92-0, Nicotinamide **1406-16-2, Vitamin D**
1406-18-4, Vitamin E 8059-24-3, Vitamin B6 12001-79-5, Vitamin K
RL: ANT (Analyte); ANST (Analytical study)
(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

IT **1406-16-2, Vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(solvent mixing device and amperometric microsensor in anal. of vitamins by **supercrit.** fluid **chromatog.**)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 14 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1997:44341 HCAPLUS

DN 126:51588

ED Entered STN: 21 Jan 1997

TI Solubilities of the Fat-Soluble Vitamins A, D, E, and K in **Supercritical Carbon Dioxide**

AU **Johannsen, Monika**; Brunner, Gerd

CS Arbeitsbereich Verfahrenstechnik II, Technical University, Hamburg, D-21073, Germany

SO Journal of Chemical and Engineering Data (**1997**), 42(1), 106-111
CODEN: JCEAAX; ISSN: 0021-9568

PB American Chemical Society

DT Journal

LA English

CC 68-1 (Phase Equilibriums, Chemical Equilibriums, and Solutions)
Section cross-reference(s): 17, 26

AB Solubilities of eight different species of the fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide** were measured at (313, 333, and 353) K and over a pressure range of 20 MPa to 35 MPa. Solubilities have been determined by an anal. method using the direct coupling of an equilibrium cell to a **supercrit.** fluid **chromatog.** system with **UV** detection. The solubilities of all fat-soluble vitamins in **supercrit. carbon dioxide** under the conditions investigated are in the range of 10 g/kg, except for β-carotene (provitamin A), which is 3 orders of magnitude less soluble With increasing mol. mass of the vitamin, its solubility in **supercrit. carbon dioxide** decreases. At constant temperature, the solubility of all substances increases with increasing d. At constant d., a rise of temperature results in an increase in solubility This is caused by the increasing vapor pressure of the solid.

ST soly fat soluble vitamin **supercrit** solvent; vitamin soly **supercrit carbon dioxide**

IT Vitamins
RL: PRP (Properties)

(fat-soluble; solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

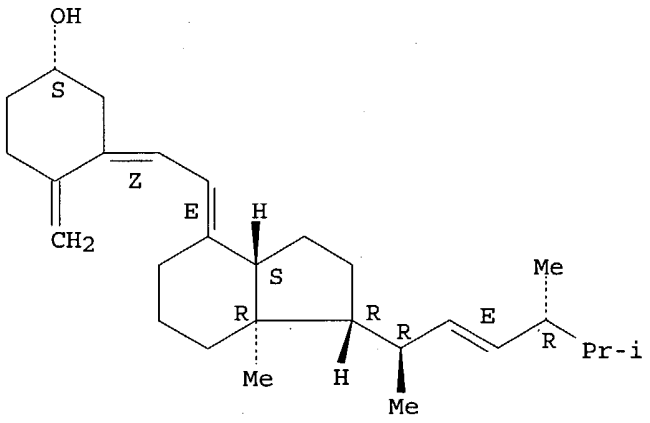
IT Solubility
(solubilities of fat-soluble vitamins A, D, E, and K in **supercrit. carbon dioxide**)

IT **50-14-6**, **Vitamin D2** 58-27-5, Vitamin K3
67-97-0, **Vitamin D3** 68-26-8, trans-Retinol
119-13-1, δ-Tocopherol **124-38-9**, **Carbon dioxide**, properties 7235-40-7, β,β-Carotene
10191-41-0, DL-α-Tocopherol 11104-38-4, Vitamin K1
RL: PRP (Properties)
(solubilities of fat-soluble **vitamins** A, D, E, and K in **supercrit. carbon dioxide**)

IT **50-14-6**, **Vitamin D2** **67-97-0**, **Vitamin D3** **124-38-9**, **Carbon dioxide**, properties
RL: PRP (Properties)
(solubilities of fat-soluble **vitamins** A, D, E, and K in **supercrit. carbon dioxide**)

RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.

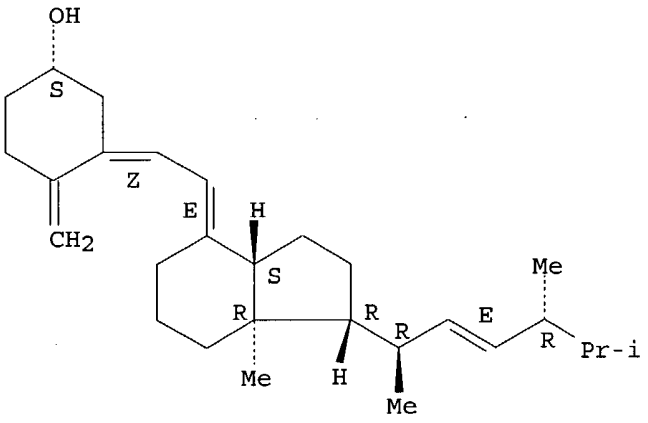
chromatog. of fat-soluble vitamins using liquid crystal polysiloxane coated particles)

IT **50-14-6, Vitamin d2 67-97-0, Vitamin d3** 1406-18-4, Vitamin e 11032-49-8, Vitamin k2 11103-57-4, Vitamin a 11104-38-4, Vitamin k1
RL: ANT (Analyte); ANST (Analytical study)
(packed capillary column **supercrit.** fluid **chromatog**. of fat-soluble **vitamins** using liquid crystal polysiloxane coated particles)

IT **50-14-6, Vitamin d2 67-97-0, Vitamin d3**
RL: ANT (Analyte); ANST (Analytical study)
(packed capillary column **supercrit.** fluid **chromatog**. of fat-soluble **vitamins** using liquid crystal polysiloxane coated particles)

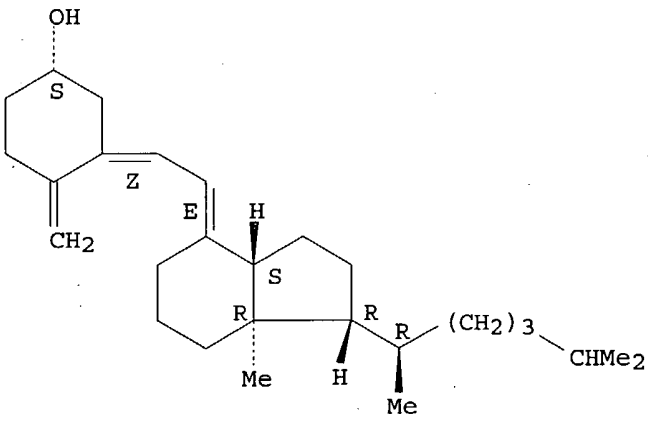
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



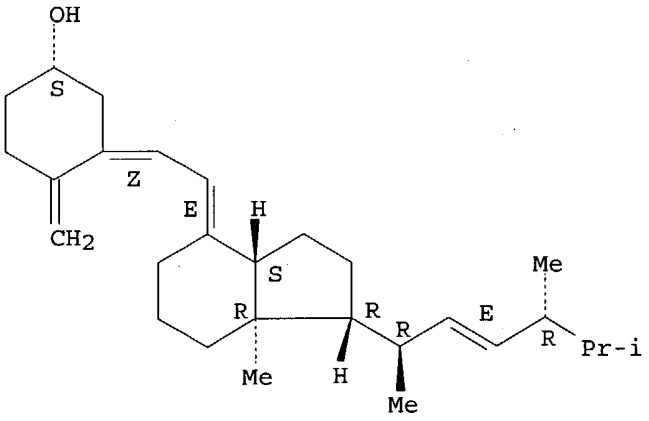
L79 ANSWER 16 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:956938 HCAPLUS
DN 124:105153
ED Entered STN: 01 Dec 1995
TI Applications of reversed-phase high performance liquid **chromatography** using enhanced-fluidity liquid mobile phases
AU Lee, Stephen T.; Olesik, Susan V.; Fields, Steven M.
CS Department of Chemistry, The Ohio State University, Columbus, OH, 43210, USA
SO Journal of Microcolumn Separations (**1995**), 7(5), 477-83
CODEN: JMSEEJ; ISSN: 1040-7685
PB Wiley
DT Journal
LA English
CC 80-4 (Organic Analytical Chemistry)
Section cross-reference(s): 51, 64
AB Enhanced-fluidity liquid mobile phases (methanol/H2O/**CO2**) were used as eluents in reversed-phase HPLC. The low pressure drop across the column allowed serial connection of micro-scale columns to achieve the efficient separation of a coal tar sample. Other applications such as the separation of fat soluble vitamins and probucol and related compds. are shown.
ST vitamin sepn reversed phase HPLC; reversed phase HPLC enhanced fluidity; coal tar reversed phase HPLC; drug analysis reversed phase HPLC; liq **chromatog** enhanced fluidity mobile phase
IT Pharmaceutical analysis
(coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)
IT Tar
RL: ANT (Analyte); ANST (Analytical study)
(coal, coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, coal tar and vitamins and drugs separation by reversed-phase high
performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)
IT **Chromatography, column and liquid**
(**high-performance reversed-phase**
, coal tar and vitamins and drugs separation by **reversed-phase high performance** liquid **chromatog.** using enhanced-fluidity liquid mobile phases)
IT Aromatic hydrocarbons, analysis
RL: ANT (Analyte); ANST (Analytical study)
(polycyclic, coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)
IT **50-14-6**, Ergocalciferol 50-32-8, (Benzo[a]pyrene), analysis 53-70-3, Dibenz[a,h]anthracene 56-55-3, Benzo[a]anthracene **67-97-0**, **Cholecalciferol** 68-26-8, trans-Retinol 71-43-2, Benzene, analysis 85-01-8, Phenanthrene, analysis 86-73-7, Fluorene 91-20-3, Naphthalene, analysis 116-31-4, trans-Retinal 120-12-7, Anthracene, analysis 127-47-9, Retinol acetate 128-37-0, BHT, analysis 128-38-1 129-00-0, Pyrene, analysis 191-24-2, Benzo[ghi]perylene 193-39-5, (Indeno[1,2,3-cd]pyrene) 205-99-2, Benzo[b]fluoranthene 206-44-0, Fluoranthene 207-08-9, (Benzo[k]fluoranthene) 208-96-8, Acenaphthylene 218-01-9, Chrysene 2455-14-3 10191-41-0 11104-38-4, Vitamin K1 23288-49-5, Probucol 26067-78-7 51571-18-7 52225-20-4 129895-82-5
RL: ANT (Analyte); ANST (Analytical study)

(coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)

IT **50-14-6**, Ergocalciferol **67-97-0**, **Cholecalciferol**
RL: ANT (Analyte); ANST (Analytical study)
(coal tar and vitamins and drugs separation by reversed-phase high performance liquid **chromatog.** using enhanced-fluidity liquid mobile phases)

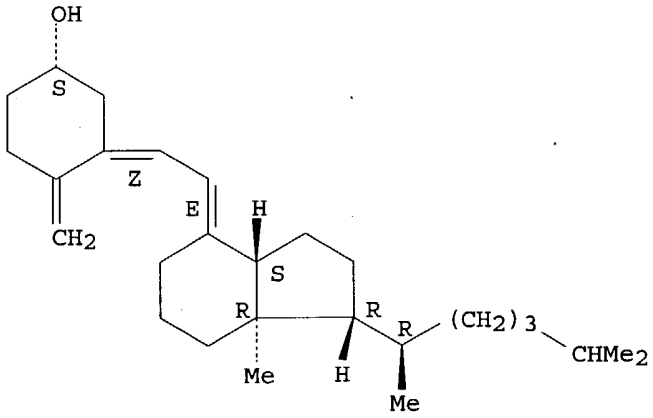
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 17 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1995:319449 HCAPLUS
DN 122:104187
ED Entered STN: 28 Jan 1995
TI Quantitative analysis of marine oils by capillary **supercritical** fluid **chromatography**

AU Staby, A.; Borch-Jensen, C.; Balchen, S.; Mollerup, J.
CS Dep. Chem. Eng., Technical Univ. Denmark, Lyngby, 2800, Den.
SO Chromatographia (**1994**), 39(11/12), 697-705
CODEN: CHRGB7; ISSN: 0009-5893
PB Vieweg
DT Journal
LA English
CC 17-1 (Food and Feed Chemistry)
AB **Supercrit.** fluid **chromatog.** anal. methods have been employed in the examination of several marine oils for the group separation of free fatty acids, retinol, ergocalciferol, **cholecalciferol**, squalene, tocopherols, cholesterol, wax esters, diacylglycerols, cholesteryl esters, and triacylglycerols. The oils were derived from characteristic species including shark, seal, edible and trash fish. The **supercrit.** fluid **chromatog.** (SFC) method used for the separation of the liqs. utilize **carbon dioxide** as the mobile phase, a non-polar capillary column, and flame ionization detection. The SFC methods have proved capable of making a considerable contribution to the continuing investigations into the structure and composition of marine oils. Furthermore SFC analyses, with their very simple sample preparation requirements, may serve as alternatives or supplements to the existing range of **chromatog.** and non-**chromatog.** anal. methods used in the examination of these oils.
ST fish oil capillary **supercrit** fluid **chromatog**; marine oil capillary **supercrit** fluid **chromatog**
IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(basking shark; determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Cod-liver oil
RL: AMX (Analytical matrix); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Fatty acids, analysis
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Glycerides, analysis
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Tocopherols
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Waxes and Waxy substances
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT **Chromatography, supercritical fluid**
(**capillary**, determination of marine oil constituents by **capillary supercrit.** fluid **chromatog.**)
IT Glycerides, analysis
RL: ANT (Analyte); ANST (Analytical study)
(di-, determination of marine oil constituents by capillary **supercrit** . fluid **chromatog.**)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)
IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)

(fish, determination of marine oil constituents by capillary **supercrit**. fluid **chromatog.**)

IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(herring, determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)

IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(mackerel, determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)

IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(sand eel, determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)

IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(seal, determination of marine oil constituents by capillary **supercrit**. fluid **chromatog.**)

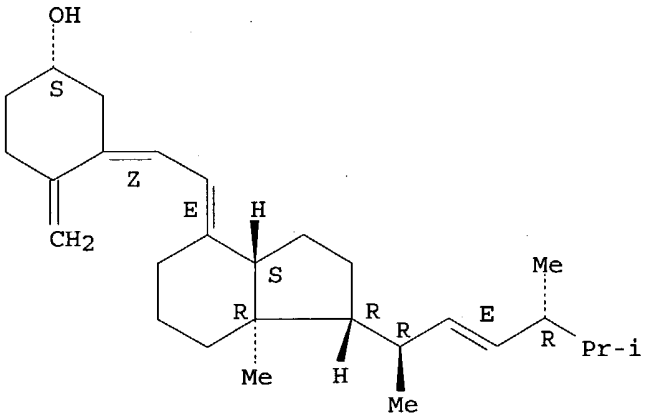
IT Fats and Glyceridic oils
RL: AMX (Analytical matrix); ANST (Analytical study)
(tuna, determination of marine oil constituents by capillary **supercrit**. fluid **chromatog.**)

IT **50-14-6**, Ergocalciferol 57-88-5, Cholesterol, analysis
57-88-5D, Cholesterol, esters **67-97-0**, **Cholecalciferol**
68-26-8, Retinol 111-02-4, Squalene 119-13-1, δ-Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)

IT **50-14-6**, Ergocalciferol **67-97-0**, **Cholecalciferol**
RL: ANT (Analyte); ANST (Analytical study)
(determination of marine oil constituents by capillary **supercrit.** fluid **chromatog.**)

RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.

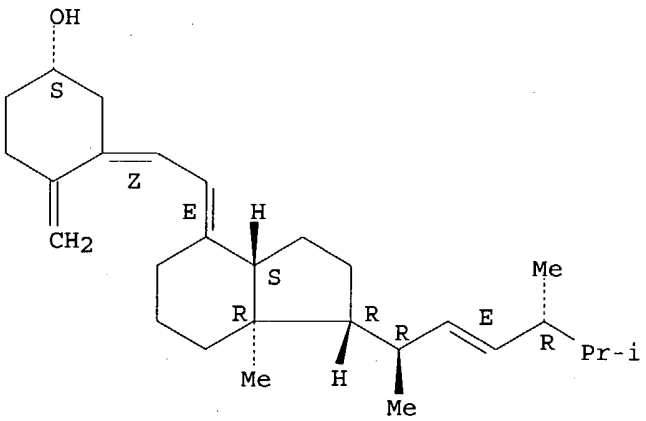


RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.

groups plays an important role.
ST **supercrit** fluid **chromatog** retention behavior; stationary phase **supercrit** fluid **chromatog**; xanthine **supercrit** fluid **chromatog**; vitamin **supercrit** fluid **chromatog**
IT **Silica gel, compounds**
RL: ANST (Analytical study)
(**alkylated**, as stationary phase in **supercrit.** fluid **chromatog.**, retention behavior of)
IT **Silica gel, compounds**
RL: ANST (Analytical study)
(**aminopropylated**, as stationary phase in **supercrit.** fluid **chromatog.**, retention behavior of)
IT **Silica gel, compounds**
RL: ANST (Analytical study)
(cyanopropylated, as stationary phase in **supercrit.** fluid **chromatog.**, retention behavior of)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, **chromatog.** of, **supercrit.**-fluid, stationary phases in)
IT **Chromatography, gas**
(**supercrit.**, of fat-soluble vitamins and xanthines)
IT **Chromatography, gas**
(**supercrit.**, **stationary phases**, silica gel derivs. as, retention behavior of)
IT **50-14-6**, **Vitamin D2** 58-08-2, Caffeine, analysis 58-55-9, Theophylline, analysis 68-26-8, Vitamin A 69-89-6D, derivs. 83-67-0, Theobromine 1406-18-4, Vitamin E
RL: ANT (Analyte); ANST (Analytical study)
(**chromatog.** of, **supercrit.**-fluid, stationary phases in)
IT **50-14-6**, **Vitamin D2**
RL: ANT (Analyte); ANST (Analytical study)
(**chromatog.** of, **supercrit.**-fluid, stationary phases in)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

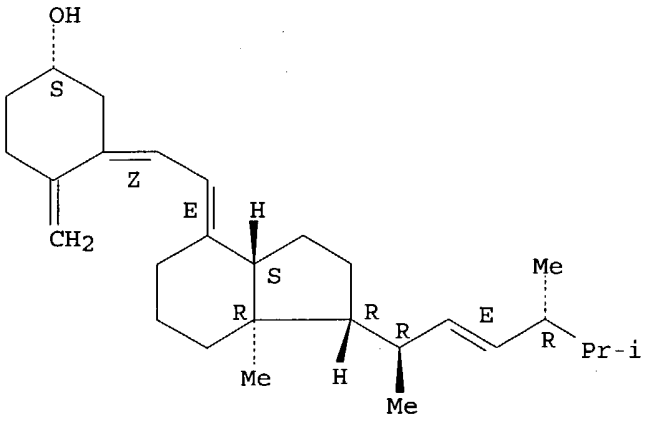
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 20 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1983:586740 HCAPLUS

DN 99:186740
ED Entered STN: 12 May 1984
TI The use of modifiers in **supercritical** fluid **chromatography** with **carbon dioxide**
AU Board, R.; McManigill, D.; Weaver, H.; Gere, D.
CS Hewlett-Packard Co., Avondale, PA, 19311, USA
SO ChemSA (**1983**), (June), 12, 21-2, 24
CODEN: CHEMDU; ISSN: 0379-4687
DT Journal
LA English
CC 80-4 (Organic Analytical Chemistry)
Section cross-reference(s): 2, 64
AB The effects of modifiers, such as MeOH, iso-PrOH, and THF, in **supercrit.** fluid **chromatog** with **CO2** as the mobile phase were studied. The retention times of di-Me terephthalate, di-Me isophthalate, and di-Me phthalate on a RP-18 bonded phase column were altered >50 times by changing the modifier concentration from 0-1% in the **CO2** mobile phase. The elution of ubiquinones and fat soluble vitamins from a reversed phase RP-18 column was possible only after the addition of MeOH to the mobile phase. With a MeOH modifier concentration of 1.5%, progesterone was separated from estrone on a reversed phase RP-8 column at 34°. At MeOH concns. >1%, the retention times of nicotine and caffeine on a polystyrene reversed phase column were affected significantly. Stationary phase-modifier interaction was the predominant mechanism for changes in retention times at modifier concns. ≤1%.
ST **supercrit** gas **chromatog** modifier effect; methanol modifier **supercrit** gas **chromatog**; isopropanol modifier **supercrit** gas **chromatog**; THF modifier **supercrit** gas **chromatog**; terephthalate dimethyl **supercrit** gas **chromatog**; isophthalate dimethyl **supercrit** gas **chromatog**; phthalate dimethyl **supercrit** gas **chromatog**; ubiquinone **supercrit** gas **chromatog**; vitamin **supercrit** gas **chromatog**; progesterone **supercrit** gas **chromatog**; estrone **supercrit** gas **chromatog**
IT **Chromatography, gas**
(**supercrit.**, with **carbon dioxide** mobile phase, modifier effects in)
IT 67-56-1, uses and miscellaneous 67-63-0, uses and miscellaneous 109-99-9, uses and miscellaneous
RL: ANST (Analytical study); USES (Uses)
(as modifier in **supercrit.** fluid **chromatog.** with **carbon dioxide** mobil phase)
IT **50-14-6** 54-11-5 57-83-0, analysis 58-08-2, analysis 68-26-8 120-61-6 131-11-3 303-95-7 303-97-9 606-06-4 1065-31-2 1406-18-4 1459-93-4 2394-68-5
RL: ANST (Analytical study)
(separation of, by **supercrit.** fluid **chromatog.** with **carbon dioxide** mobile phase, modifier effects on)
IT **50-14-6**
RL: ANST (Analytical study)
(separation of, by **supercrit.** fluid **chromatog.** with **carbon dioxide** mobile phase, modifier effects on)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



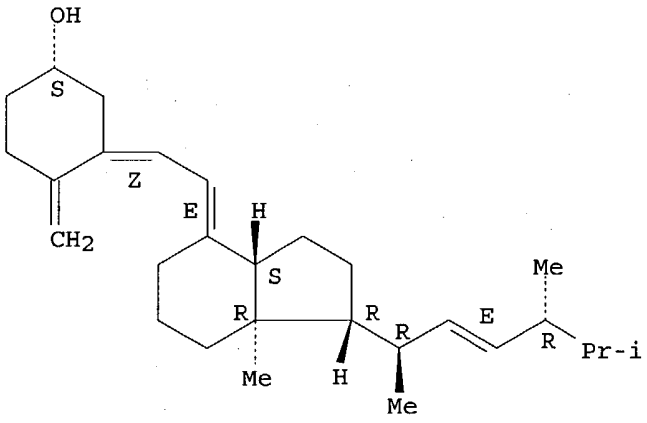
L79 ANSWER 21 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1981:495426 HCAPLUS
DN 95:95426
ED Entered STN: 12 May 1984
TI Possibility of using the **carbon dioxide** extract of yeast residues of grape wines for cosmetics
AU Kupriyanova, L. A.; Shcherbakov, V. G.; Kalistratova, T. P.; Pekhov, A. V.; Cherevatyi, V. S.; Lebedeva, A. I.
CS Krasnodar. Nauchno-Issled. Inst. Pishchevoi Prom., Krasnodar, USSR
SO Izvestiya Vysshikh Uchebnykh Zavedenii, Pishchevaya Tekhnologiya (**1981**), (3), 40-2
CODEN: IVUPA8; ISSN: 0579-3009
DT Journal
LA Russian
CC 16-3 (Fermentations)
Section cross-reference(s): 62
AB The **CO2** extract of wine lees contains 1% **provitamin D** (calciferol), 190 mg% provitamin A (carotenoid), 30 mg% vitamin E (tocopherol), and 35% essential fatty acids. Palmitate and linoleate constitute 32.42 and 34.38%, resp., of the total fatty acid content. The **CO2** extract maintains its biol. activity at 20° in the dark for 6 mo and may be used as a supplement in the cosmetic industry.
ST wine yeast ext cosmetics; **carbon dioxide** yeast ext cosmetics
IT Cosmetics
(**carbon dioxide** extract of wine yeast in)
IT Fatty acids, biological studies
Vitamins
RL: BIOL (Biological study)
(of yeast extract, cosmetics in relation to)
IT Yeast
(wine, **carbon dioxide** extract of, as cosmetics ingredient)
IT **124-38-9**, biological studies
RL: BIOL (Biological study)
(yeast extract of, as cosmetics ingredient)
IT **124-38-9**, biological studies
RL: BIOL (Biological study)
(yeast extract of, as cosmetics ingredient)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

(123) Tondeur, D; Ind Eng Chem Res 1995, V34, P2782 HCAPLUS
(124) Unger, K; Handbuch der HPLC 1994
(125) Upnmoor, D; Dissertation, Technische Universitat Hamburg-Harburg 1992
(126) Verillon, F; Supercritical Fluid Chromatography with Packed Columns: Techniques and Applications 1998
(127) Villeneuve, M; Analytica 2000:9th International and 4th European Symposium on Supercritical Fluid Chromatography 2000
(128) Whatley, J; J Chromatogr A 1995, V697(12), P251
(129) White, C; Analysis of Pharmaceutical and Other Solutes of Biochemical Importance by Supercritical Fluid Chromatography 1988, V11, P94 HCAPLUS
(130) Wilson, J; J Am Chem Soc 1940, V62, P1583 HCAPLUS
(131) Yakushina, L; J Pharmaceutical & Biomedical Analysis 1995, V13(4/5), P715 HCAPLUS
(132) Yamauchi, Y; J Chromatogr 1990, V505, P237 HCAPLUS

IT **50-14-6**, **Vitamin D2 67-97-0**, **Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(preparative **supercrit.** fluid **chromatog.** as an example of **vitamin** separation)

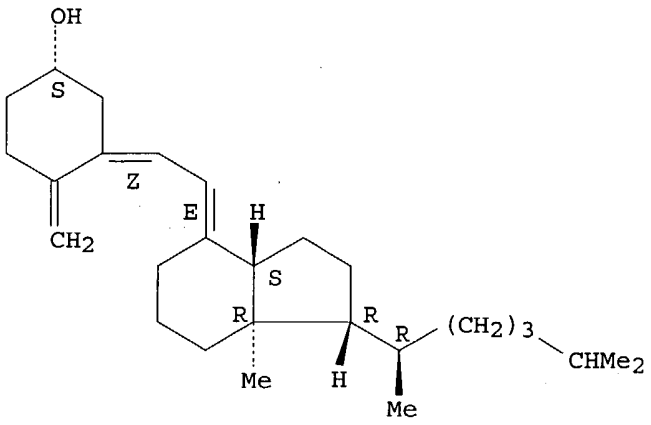
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 5 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2002:593413 HCAPLUS
DN 137:278027
ED Entered STN: 09 Aug 2002
TI Development of methods for the determination of vitamins A, E and β-carotene in processed foods based on **supercritical** fluid extraction: a collaborative study
AU Mathiasson, L.; Turner, C.; Berg, H.; Dahlberg, L.; Theobald, A.; Anklam, E.; Ginn, R.; Sharman, M.; Ulberth, F.; Gabernig, R.
CS Department of Analytical Chemistry, Lund University, Lund, S-221 00, Swed.
SO Food Additives and Contaminants (2002), 19(7), 632-646
CODEN: FACOEB; ISSN: 0265-203X
PB Taylor & Francis Ltd.
DT Journal
LA English
CC 17-1 (Food and Feed Chemistry)
AB New methodologies based on **supercrit.** fluid extraction (SFE) have been developed for the determination of fat-soluble vitamins in processed foods. The results obtained so far indicate that SFE is well suited to extraction of fat-soluble vitamins from food products, although validation work is required to establish accuracy and precision. The vitamins investigated were A, E and β-carotene, and the processed foods were UHT milk, milk powder, minced meat, liver paste, infant formula, canned baby food and margarine. Extraction equipment employed analyte collection on either a solid-phase trap or in a solvent. After extraction, the samples were saponified and the vitamins determined using reversed-phase liquid **chromatog.** with **UV** or fluorescence detection. Sample throughput was at least 12 samples day-1, i.e. at least twice the number achievable with a conventional extraction methodol. The detection limits for the vitamins in different processed foods were well below 0.1 μg g-1. Recoveries (in comparison with vitamin levels obtained using conventional solvent extraction) were close to 100% for experienced personal with access to modern automatic equipment. To reach this level, it was necessary to protect the vitamins with an antioxidant during the different steps of the anal. procedure, to add methanol or ethanol to the extraction cell to facilitate the analyte extraction from the food matrix, and when using a solid-phase trap, to employ a fractionated extraction-elution procedure to prevent breakthrough losses. The developed methods were tested in a validation exercise between five labs., which had taken part in the method development, and in an intercomparison between 10 labs. including labs. with less experience of vitamin determination The

within-laboratory RSD was generally ≤11%. The average of the between-laboratory relative standard deviation (RSD) was about 23% in the validation, and increased to about 40% in the intercomparison. Ruggedness tests performed at different steps of the project showed that different types and models of equipment did not give large differences in recoveries. Thus, the increasing RSD can largely be ascribed to differences in experience in vitamin anal. of the participants.

ST vitamin detn food **supercrit** fluid extn; carotene detn food **supercrit** fluid extn

IT Food analysis
Margarine
Milk analysis
Reversed phase HPLC
Sample preparation
Saponification
(determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Milk substitutes
(human; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Canned foods
(infant; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Meat
(liver, paste; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Meat
(pork; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT Extraction
(**supercrit.**; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT 50-81-7, Ascorbic acid, uses
RL: ARG (Analytical reagent use); ANST (Analytical study); USES (Uses)
(antioxidant; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT **50-14-6**, **Vitamin D2** 59-02-9, α-Tocopherol **67-97-0**, **Vitamin D3** 68-26-8, all-trans-Retinol 119-13-1, δ-Tocopherol 148-03-8, β-Tocopherol 1406-18-4, Vitamin E 7235-40-7, β-Carotene 7616-22-0, γ-Tocopherol 11103-57-4, Vitamin A
RL: ANT (Analyte); ANST (Analytical study)
(determination of **vitamins** A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT **124-38-9**, **Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses
RL: ARG (Analytical reagent use); ANST (Analytical study); USES (Uses)
(modifier; determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

RE.CNT 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

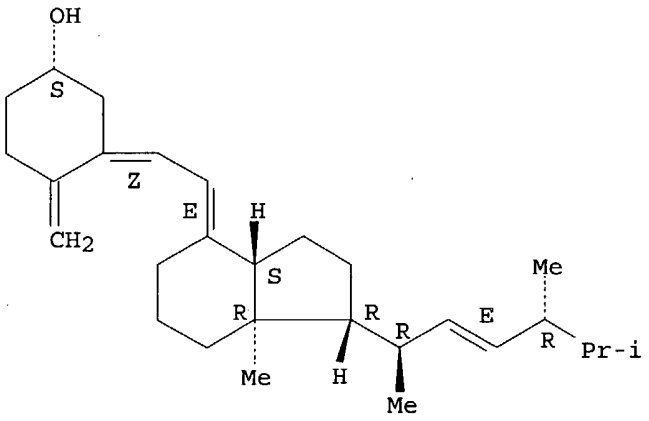
(1) AOAC; Liquid Chromatographic Method. Final Action 1993, P555
(2) AOAC; Liquid Chromatographic Method. Final Action 1993, P580
(3) AOAC; Liquid Chromatographic Method. First Action 1993, P591
(4) Anon; Dairy Chemistry and Physics 1984

(5) Berg, H; Journal of Biochemical and Biophysical Methods 2000, V43, P391 HCAPLUS
(6) Bjorklund, E; Journal of Liquid Chromatography and Related Technologies 2001, V24(14), P2133 HCAPLUS
(7) Bui, M; Journal of the Association of Official Analytical Chemistry 1987, V70, P802 HCAPLUS
(8) Burri, B; Journal of Chromatography A 1997, V762, P201 HCAPLUS
(9) CEN; European Standard Method 1997, TC275/WG9N34, PREN12823
(10) CEN; European Standard Method 1997, TC275/WG9N36
(11) CEN; European Standard Method 1997, TC275/WG9N33, PREN12822
(12) CEN; European Standard Method 1997, TC275/WG9N34, PREN12823
(13) Chow, F; Lipids 1983, V18, P837 HCAPLUS
(14) De Vries, E; Journal of the Association of Official Analytical Chemistry 1982, V65, P1228 HCAPLUS
(15) Gregory, J; Food Chemistry 1996, P531 HCAPLUS
(16) Hollman, P; Analyst 1993, V118, P475 HCAPLUS
(17) Indyk, H; New Zealand Journal of Dairy Science and Technology 1985, V20, P19 HCAPLUS
(18) May, W; Standard Reference Material 1846 Infant formula 1999
(19) Panfili, G; Analyst 1994, V119, P1161 HCAPLUS
(20) Reynolds, S; Analyst 1984, V109, P489 HCAPLUS
(21) Schneiderman, M; Journal of Chromatography A 1997, V765, P215 HCAPLUS
(22) Schneiderman, M; Journal of the Association of Official Analytical Chemistry 1988, V71, P815 HCAPLUS
(23) Spanos, G; Journal of Food Science 1993, V4, P817
(24) Tanner, J; Journal of the Association of Official Analytical Chemistry International 1993, V76, P399 HCAPLUS
(25) Turner, C; Journal of Agricultural and Food Chemistry 2001, V49, P553 HCAPLUS
(26) Turner, C; Journal of Chromatography A 2000, V874, P275 HCAPLUS
(27) Zamarreno, M; Journal of Chromatography 1992, V623, P69
(28) Zonta, F; Journal of Chromatography 1982, V246, P105 HCAPLUS

IT **50-14-6, Vitamin D2 67-97-0, Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(determination of **vitamins** A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)

RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

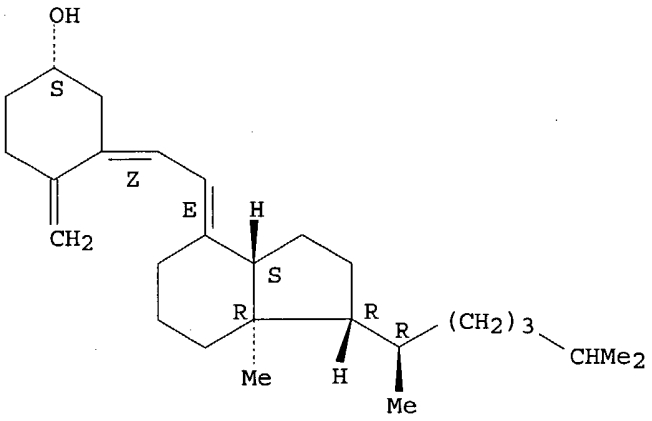
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



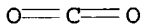
RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX

NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT **124-38-9**, **Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(determination of vitamins A, E and β-carotene in processed foods based on **supercrit.** fluid extraction)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 6 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2001:780606 HCAPLUS
DN 135:303109
ED Entered STN: 26 Oct 2001
TI Method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercritical CO2** and by introducing desired lipophilic substances using compressed **CO2**
IN Heidlas, Juergen; Stork, Kurt; Zhang, Zhengfeng; Ober, Martin; Wiesmueller, Johann; Obersteiner, Johann
PA Degussa A.-G., Germany
SO PCT Int. Appl., 23 pp.
CODEN: PIXXD2
DT Patent
LA German
IC ICM A23L001-015
ICS A23K001-16; A23G001-02; A23G001-00; A23L001-10; A23L001-164
CC 17-4 (Food and Feed Chemistry)
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI WO 2001078525	A1	20011025	WO 2001-EP3984	20010406

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,

BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG
DE 10018606 A1 20011025 DE 2000-10018606 20000414
EP 1272051 A1 20030108 EP 2001-940300 20010406
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
PRAI DE 2000-10018606 A 20000414
WO 2001-EP3984 W 20010406

AB The invention relates to a method for producing foods and/or feeds having improved physiol. properties by using dry compressed gases. According to the invention, lipid constituents, in particular triglycerides and other fat accompanying substances, are removed from the food matrix by means of extraction involving the use of **supercrit. CO2** optionally having a proportion of up to 50 weight% propane. The extracted lipid constituents are then separated using known methods, and the extracted food matrix lipophilic constituents, particularly nutrition-physiol. enhancing additives, are homogeneously introduced using compressed **CO2** optionally having a proportion of up to 50 weight% propane. The particular implementation of the method at preferred pressures ranging from 100 to 800 bar and at temps. ranging from 31 to 100°C enables the specific introduction of, in particular, fat-soluble vitamins, their derivs. or precursors as well as polyunsatd. fatty acids, lipoic acids or sterol derivs. In this manner, foods or feeds can be obtained such as cereals with polyunsatd. fatty acids, cocoa and egg-yolk products which, in the form of functional foods, take the modified nutritional properties into account.

ST food feed processing lipid **supercrit carbon dioxide**

IT Vitamins
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(fat-soluble; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Egg yolk
(fats; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Cereal (grain)
Food functional properties
Lipophilicity
Oatmeal
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Cocoa butter
Sterols
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Fats and Glyceridic oils, processes
RL: REM (Removal or disposal); PROC (Process)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Glycerides, processes
RL: REM (Removal or disposal); PROC (Process)
(method for producing foodstuffs and/or feedstuffs by removing

process); BIOL (Biological study); PROC (Process); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT **124-38-9**, **Carbon dioxide**, biological studies
RL: FFD (Food or feed use); PEP (Physical, engineering or chemical process); BIOL (Biological study); PROC (Process); USES (Uses)
(**supercrit.**; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

RE.CNT 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE

(1) Commanditaire Vennootschap Op; GB 340580 A 1930 HCAPLUS
(2) Cully, J; US 5061505 A 1991 HCAPLUS
(3) Gen Foods Inc; EP 0554993 A 1993
(4) Heidlas, J; US 5616352 A 1997
(5) Jackeschky, M; WO 9314649 A 1993
(6) McLachlan, C; US 5024846 A 1991 HCAPLUS
(7) Mitsubishi Corp; EP 0531104 A 1993 HCAPLUS
(8) Nicol, R; US 5147672 A 1992
(9) Royal Society Of Chemistry; MCCANCE & WIDDOWSON'S: "The Composition of Foods - 5th Ed" 1992, P40
(10) Vecsei, R; DE 3540544 A 1987

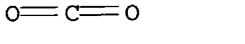
IT **1406-16-2**, **Vitamin D**
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT **124-38-9**, **Carbon dioxide**, biological studies
RL: FFD (Food or feed use); PEP (Physical, engineering or chemical process); BIOL (Biological study); PROC (Process); USES (Uses)
(**supercrit.**; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

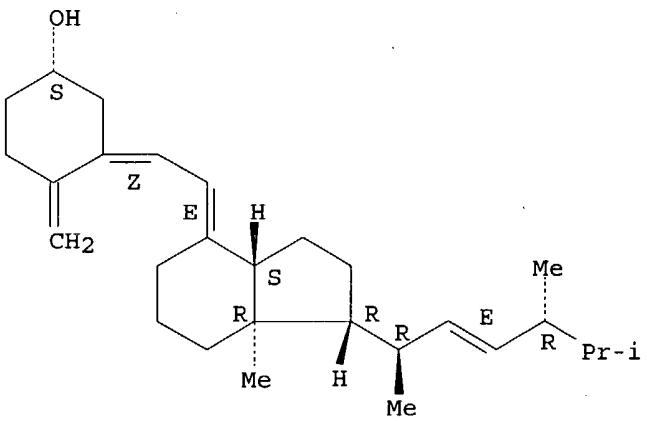


L79 ANSWER 7 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2001:317898 HCAPLUS
DN 135:200389
ED Entered STN: 04 May 2001
TI Phase equilibria of the **vitamins D2**, **D3** and K3 in binary systems with **CO2** and propane
AU Knez, Z.; Skerget, M.
CS Faculty of Chemistry and Chemical Engineering, University of Maribor, Maribor, SI-2000, Slovenia
SO Journal of Supercritical Fluids (2001), 20(2), 131-144
CODEN: JSFLEH; ISSN: 0896-8446
PB Elsevier Science B.V.
DT Journal

LA English
CC 63-8 (Pharmaceuticals)
Section cross-reference(s): 68
AB Solubilities of the fat-soluble **vitamins** K3, **D3** and D2 in **CO2** and propane were measured at temps. 30, 40, 60 and 80°C and over a pressure range of 80-300 bar for **CO2** and 10-110 bar for propane using a static-analytic method. In order to verify weather, the solubility isotherm crosses the three-phase S-L-V line, solid-liquid (S-L) phase transitions of **vitamins** K3, **D3** and D2 under the pressure of gas were determined with modified capillary method. For all **vitamins** under the pressure of **CO2**, propane and di-Me ether, a neg. dP/dT slope of the S-L-V curve was found. For K3 under the pressure of inert gas (argon and nitrogen), the S-L-V-curve had a pos. dP/dT slope. The solubilities of **vitamins D** in dense **CO2** under the conditions investigated were in the range 0.04+10-3-1.45+10-3 mole fraction. For **vitamin** K3 the solubility in **CO2** was higher and was in the range 0.16+10-3-4.07+10-3 mole fraction. The solubilities of **vitamins D** in propane were approx. up to 10 times higher as in **CO2** and in the range from 0.15+10-3 to 12.40+10-3 mole fraction. Opposite, the solubility of **vitamin** K3 in propane was lower than in **CO2** and was in the range from 0.03+10-3 to 2.99+10-3 mole fraction. The exptl. S-L-V data were correlated using Peng-Robinson equation of state and binary iteration parameters obtained from best fit were used for solubility calcn. The fit of S-L-V lines showed good results in the case of **vitamin** K3 and the order of magnitude of the solid solubilities were well represented.
ST calciferol soly **carbon dioxide** propane pressure; **cholecalciferol** soly **carbon dioxide** propane pressure; vitamin K3 soly **carbon dioxide** propane pressure
IT **Vitamins**
RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(fat-soluble; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)
IT Pressure
(gas; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)
IT Binary systems
Phase transition
Solid solubility
(phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)
IT Extraction
(**supercrit.**; phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane for high pressure separation)
IT **50-14-6, vitamin D2** 58-27-5, **vitamin K3** **67-97-0, vitamin D3**
RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)
IT 74-98-6, Propane, properties **124-38-9, Carbon dioxide**, properties
RL: PRP (Properties)
(phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2** and propane)
IT 7440-37-1, Argon, properties 7727-37-9, Nitrogen, properties
RL: PRP (Properties)
(phase equilibrium of **vitamins D2, D3** and K3 in binary systems with **CO2**, propane, di-Me ether, argon, or

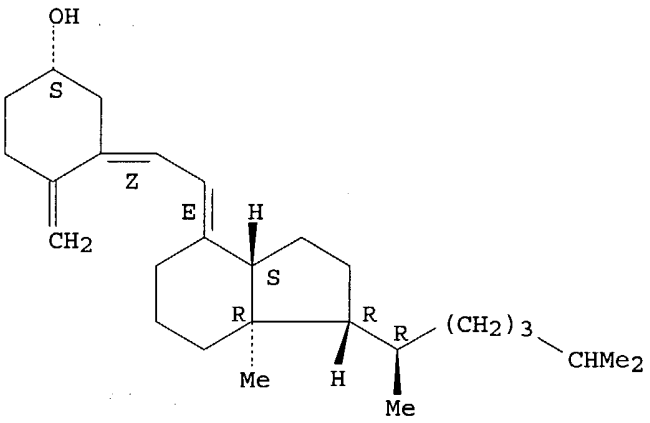
nitrogen)
IT 115-10-6, Dimethylether
RL: PRP (Properties)
(phase equilibrium of **vitamins D2, D3** and K3
in binary systems with **CO2**, propane, or di-Me ether)
RE.CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; ASPEN MAX Release 1.1
(2) Baum, E; Chemical Property Estimation. Theory and Application 1998
(3) Birtigh, A; J Supercrit Fluids 1995, V8, P46 HCAPLUS
(4) Chrastil, J; J Phys Chem 1982, V86, P3016 HCAPLUS
(5) Cygnarowicz, M; Fluid Phase Equilib 1990, V59, P57 HCAPLUS
(6) Jay, A; J Supercrit Fluids 1992, V5, P274 HCAPLUS
(7) Johannsen, M; J Chem Eng Data 1997, V42, P106 HCAPLUS
(8) Knez, Z; Fluid Phase Equilib 1998, V152, P95 HCAPLUS
(9) Knez, Z; J Supercrit Fluids 1992, V5(4), P251 HCAPLUS
(10) Kokot, K; Accepted for publication in Fluid Phase Equilib
(11) McHugh, M; Supercritical Fluid Extraction. Principle and Practice 1986
(12) Meier, U; Fluid Phase Equilib 1994, V92, P289 HCAPLUS
(13) Mendes, R; J Supercrit Fluid 1999, V16(2), P99 HCAPLUS
(14) Ohgaki, K; Ind Chem Eng 1989, V29, P2
(15) Pereira, P; Fluid Phase Equilib 1993, V91, P133 HCAPLUS
(16) Sakaki, K; J Chem Eng Data 1992, V37, P249 HCAPLUS
(17) Schaffner, D; J Supercrit Fluids 1995, V8, P287 HCAPLUS
(18) Schneider, G; Proceedings of the Third International Symposium of High Pressure Chemical Engineering 1996, P259 HCAPLUS
(19) Skerget, M; Fluid Phase Equilib 1995, V109, P131 HCAPLUS
(20) Subra, P; Fluid Phase Equilib 1997, V131(1-2), P269 HCAPLUS
(21) Tuma, D; Dissertation, Fakultat fur Chemie der Ruhr-Universitat Bochum 1999
(22) Tuma, D; Fluid Phase Equilib 1999, V158-160, P743 HCAPLUS
(23) Weber, W; Proceedings of the Fifth International Symposium on Supercritical Fluids 2000
(24) Weidner, E; J Supercrit Fluids 1997, V10, P139 HCAPLUS
IT **50-14-6, vitamin D2 67-97-0, vitamin D3**
RL: PEP (Physical, engineering or chemical process); PRP (Properties); THU (Therapeutic use); BIOL (Biological study); PROC (Process); USES (Uses)
(phase equilibrium of **vitamins D2, D3** and K3
in binary systems with **CO2** and propane)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.

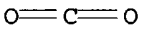


RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT **124-38-9**, **Carbon dioxide**, properties
RL: PRP (Properties)
(phase equilibrium of **vitamins D2**, **D3** and K3 in binary systems with **CO2** and propane)
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



L79 ANSWER 8 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2000:659344 HCAPLUS
DN 134:152447
ED Entered STN: 20 Sep 2000
TI Phase behavior of sterols and vitamins in **supercritical CO2**
AU Gerszt, R.; Pessoa, F. L. P.; Mendes, M. F.
CS Escola de Quimica (UFRJ), Centro de Tecnologia, Cidade Universitaria, Rio de Janeiro, CEP 21949-000, Brazil
SO Brazilian Journal of Chemical Engineering (2000), 17(3), 261-270
CODEN: BJCEFZ; ISSN: 0104-6632
PB Associacao Brasileira de Engenharia Quimica
DT Journal
LA English
CC 63-4 (Pharmaceuticals)
AB Extraction with **supercrit.** solvents has been used in different areas, such as petroleum deasphaltation, decaffeination of coffee and tea and in the separation of other types of natural products. The **supercrit.** solvent most frequently utilized in the extraction of natural products is **carbon dioxide** (**CO2**) due to its several advantages over other solvents such as low cost, non-toxicity and volatility. The design, evaluation and optimization of a **supercrit.** extraction that is based on phase equilibrium require phase equilibrium data. This type of data is very scarce for natural compds. like sterols and vitamins. These natural compds. are produced synthetically, but nowdays interest in their extraction from natural sources is increasing.

Therefore, the objective of this work is to study the thermodn. modeling equilibrium of systems containing vitamins A, D, E and K, using the predictive LCVM model. The sensitivity of critical properties in the calcn. of the phase behavior was also studied. This study proved that the choice of a group contribution method to calculate thermodn. properties is very important for obtaining good results in the phase equilibrium calcns.

ST sterol vitamin **supercrit carbon dioxide** extn; phase behavior sterol vitamin **supercrit** extn

IT Simulation and Modeling, physicochemical
(phase behavior of sterols and vitamins in **supercrit. CO2** extraction)

IT Sterols
Vitamins
RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)
(phase behavior of sterols and vitamins in **supercrit. CO2** extraction)

IT Extraction
(**supercrit.**; phase behavior of sterols and vitamins in **supercrit. CO2** extraction)

IT **124-38-9, Carbon dioxide**, uses
RL: NUU (Other use, unclassified); USES (Uses)
(phase behavior of sterols and vitamins in **supercrit. CO2** extraction)

IT **50-14-6P, Vitamin D2** 59-02-9P, α-Tocopherol **67-97-0P, Vitamin D3** 119-13-1P, δ-Tocopherol 1406-18-4P, Vitamin E 11103-57-4P, Vitamin A 11104-38-4P, Vitamin K1
RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)
(phase behavior of sterols and **vitamins** in **supercrit** . **CO2** extraction)

RE.CNT 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD

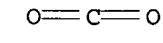
RE

(1) Birtigh, A; The Journal of Supercritical Fluids 1995, V8, P46 HCAPLUS
(2) Boukouvalas, C; Fluid Phase Equilibria 1994, V92, P75 HCAPLUS
(3) Chrastil, J; J Phys Chem 1982, V86, P3016 HCAPLUS
(4) Conn, E; Outlines of Biochemistry 5/E 1987
(5) Huron, M; Fluid Phase Equilibria 1979, V3, P255 HCAPLUS
(6) Johannsen, M; J Chem Eng Data 1997, V42, P106 HCAPLUS
(7) Meier, U; Fluid Phase Equilibria 1994, V92, P289 HCAPLUS
(8) Melo, S; Brazilian Journal of Chemical Engineering 1996, V13(04), P192
(9) Michelsen, M; Fluid Phase Equilibria 1990, V60, P213 HCAPLUS
(10) Ohgaki, K; International Chemical Engineering 1989, V29(2), P302
(11) Peng, D; Ind Eng Chem Fundam 1976, V15(1), P59 HCAPLUS
(12) Pereira, P; Fluid Phase Equilibria 1993, V91, P133 HCAPLUS

IT **124-38-9, Carbon dioxide**, uses
RL: NUU (Other use, unclassified); USES (Uses)
(phase behavior of sterols and vitamins in **supercrit. CO2** extraction)

RN 124-38-9 HCAPLUS

CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



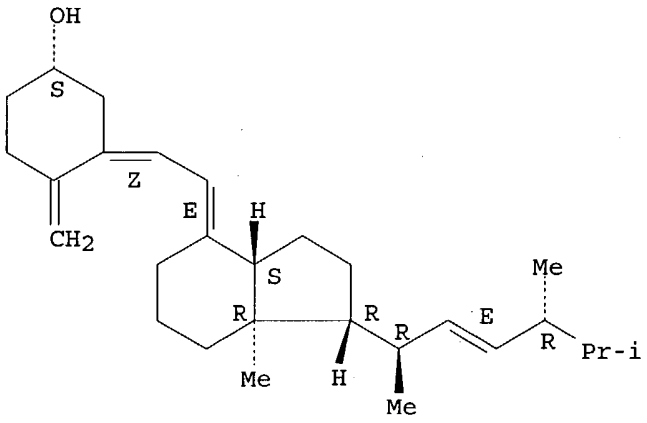
IT **50-14-6P, Vitamin D2 67-97-0P, Vitamin D3**
RL: PRP (Properties); PUR (Purification or recovery); PREP (Preparation)
(phase behavior of sterols and **vitamins** in **supercrit** . **CO2** extraction)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI)

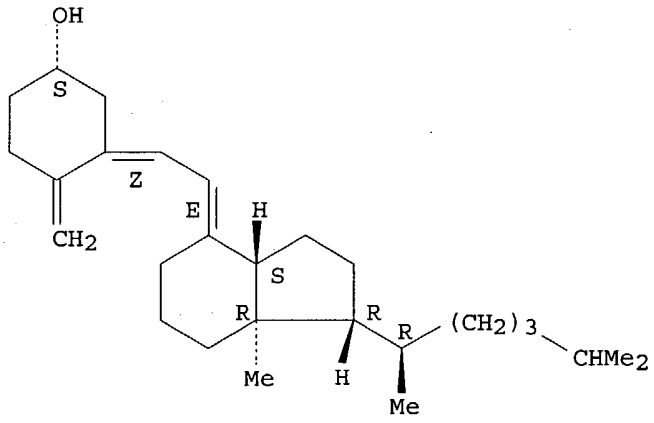
(CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 9 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2000:436772 HCAPLUS
DN 133:140337
ED Entered STN: 29 Jun 2000
TI Separation of vitamins by **supercritical** fluid **chromatography** with water-modified **carbon dioxide** as the mobile phase
AU Pyo, D.
CS Department of Chemistry, Kangwon National University, Chuncheon, 200-701, S. Korea
SO Journal of Biochemical and Biophysical Methods (2000), 43(1-3), 113-123
CODEN: JBBMDG; ISSN: 0165-022X
PB Elsevier Science Ireland Ltd.
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)

AB **Supercrit.** fluid **chromatog.** (SFC) has become a technique for solving problems that are difficult to be monitored by other **chromatog.** methods. However, the most widely used fluid, is no more polar than hexane. Polar samples which are difficult to analyze with pure **supercrit. CO2** because of their high polarity can be separated by adding polar modifiers to **supercrit. CO2.** In this study, various vitamins were well separated using water-modified **supercrit. CO2** fluid. The amount of water dissolved in **supercrit. CO2** was measured using an amperometric microsensor made of a thin film of perfluorosulfonate ionomer (PFSI).

ST vitamin sepn **supercrit** fluid **chromatog; carbon dioxide** mobile phase **chromatog** vitamin sepn

IT **Supercritical fluid chromatography**
(separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT 50-81-7, Ascorbic acid, analysis 59-43-8, Vitamin B1, analysis 59-67-6, Nicotinic acid, analysis 83-88-5, Vitamin B2, analysis 98-92-0, Nicotinamide **1406-16-2, Vitamin D** 1406-18-4, Vitamin E 8059-24-3, Vitamin B6 12001-79-5, Vitamin K
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(**supercrit.**; separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

RE.CNT 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD

RE
(1) Blilie, A; Anal Chem 1985, V57, P2239 HCAPLUS
(2) Engelhardt, H; J Chromatogr 1989, V477, P169 HCAPLUS
(3) Huang, H; Anal Chem 1990, V62, P1935 HCAPLUS
(4) Jentoft, R; J Chromatogr Sci 1970, V8, P138 HCAPLUS
(5) Novotny, M; J Chromatogr 1971, V61, P17 HCAPLUS
(6) Pyo, D; Anal Chim Acta 1993, V280, P103 HCAPLUS
(7) Pyo, D; Anal Sci 1994, V10, P171 HCAPLUS
(8) Pyo, D; Anal Sci Technol 1997, V10, P131 HCAPLUS
(9) Pyo, D; Analyst (London) 1993, V118, P253 HCAPLUS
(10) Schmidt, S; Chromatographia 1988, V25, P775 HCAPLUS
(11) Schwartz, H; Fresenius' Z Anal Chem 1988, V330, P204 HCAPLUS
(12) Shen, Y; Chromatographia 1996, V43, P53 HCAPLUS
(13) Wright, B; J Chromatogr 1986, V355, P367 HCAPLUS
(14) Yonker, C; J Chromatogr 1986, V361, P25 HCAPLUS
(15) Yonker, C; J Phys Chem 1986, V90, P3022 HCAPLUS

IT **1406-16-2, Vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT **124-38-9, Carbon dioxide**, analysis
RL: ARU (Analytical role, unclassified); ANST (Analytical study)
(**supercrit.**; separation of vitamins by **supercrit.** fluid **chromatog.** with water-modified **carbon dioxide** as mobile phase)

RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)

O=C=O

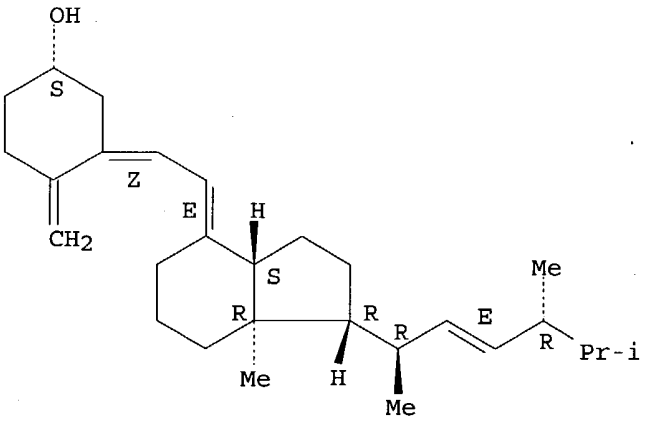
L79 ANSWER 10 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2000:311007 HCAPLUS
DN 132:339446
ED Entered STN: 14 May 2000
TI Determination of **vitamins D2** and **D3** in pharmaceuticals by **supercritical**-fluid extraction and HPLC separation with **UV** detection
AU Gamiz-Gracia, L.; Jimenez-Carmona, M. M.; Luque de Castro, M. D.
CS Analytical Chemistry Division, Faculty of Sciences, University of Cordoba, Cordoba, 14004, Spain
SO Chromatographia (2000), 51(7/8), 428-432
CODEN: CHRGB7; ISSN: 0009-5893
PB Friedrich Vieweg & Sohn Verlagsgesellschaft mbH
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)
AB The **supercrit.**-fluid extraction of **vitamins D2** and **D3** with **carbon dioxide** is reported for the first time. The extraction recovery was enhanced by direct addition of di-Et ether to sample contained in the extraction cell. Separation and detection of the analytes was performed off-line by reversed-phase HPLC with **UV** -detection. The quantification limit of the method is 4.1 μg for both analytes, with precision, expressed as relative standard deviation, of 3.8 and 6.3% for **vitamins D2** and **D3**, resp. The proposed method was applied to the determination of **vitamin D** in different pharmaceutical products; recoveries were between 85 and 105%.
ST **vitamin D** detn **supercrit** fluid extn; HPLC
vitamin D detn **supercrit** extn; liq **chromatog vitamin D** detn pharmaceutical
IT **Reversed phase HPLC**
(determination of **vitamins D2** and **D3** in pharmaceuticals by **supercrit.**-fluid extraction and HPLC with **UV** detection)
IT Extraction
(**supercrit.**; determination of **vitamins D2** and **D3** in pharmaceuticals by **supercrit.**-fluid extraction and HPLC with **UV** detection)
IT **50-14-6**, **Vitamin D2 67-97-0**, **Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(determination of **vitamins D2** and **D3** in pharmaceuticals by **supercrit.**-fluid extraction and HPLC with **UV** detection)
RE.CNT 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Adachi, T; Anal Sci 1994, V10, P457 HCAPLUS
(2) Amin, M; J Liquid Chromatogr 1988, V11, P1347 HCAPLUS
(3) Ballesteros, E; Chromatographia 1995, V40, P425 HCAPLUS
(4) Burri, B; J Chromatogr 1997, V762, P201 HCAPLUS
(5) Buskov, S; J Chromatogr 1998, V802, P233 HCAPLUS
(6) Colombo, M; Phytochem Anal 1998, V9, P192 HCAPLUS
(7) Cuadros, L; Anal Lett 1993, V26, P1243
(8) Garcia, A; Trends Anal Chem 1997, V16, P381
(9) Hart, J; Analyst 1992, V117, P1441 HCAPLUS
(10) Hondo, T; Bunseki Kagaku 1986, V35, P316 HCAPLUS

(11) Karlsson, L; J Pharm Biomed Anal 1997, V15, P601 HCAPLUS
(12) Li, K; J Chromatogr Sci 1994, V32, P53 HCAPLUS
(13) Luque de Castro, M; Analytical Supercritical-Fluid Extraction 1994
(14) Masuda, M; Anal Sci 1993, V9, P29 HCAPLUS
(15) Ortiz-Boyer, F; Chromatographia 1998, V47, P367 HCAPLUS
(16) Ortiz-Boyer, F; Clinica Chimica Acta 1998, V274, P139 HCAPLUS
(17) Royal Pharmaceutical Society Of Great Britain; Martindale, The Extra Pharmacopoeia, 29th Edn 1989, P1281
(18) Salvador, A; Anal Commun 1998, V35, P53 HCAPLUS
(19) Savolainen, K; J Pharm Sci 1988, V77, P802 HCAPLUS
(20) Scalia, S; J Pharm Biomed Anal 1995, V13, P273 HCAPLUS
(21) Scalia, S; J Pharm Sci 1995, V84, P433 HCAPLUS
(22) Schneiderman, M; J Chromatogr 1997, V765, P215 HCAPLUS
(23) Schneiman, M; J Assoc Off Anal Chem 1988, V71, P815
(24) White, C; J High Resol Chromatogr 1988, V11, P94 HCAPLUS

IT **50-14-6**, **Vitamin D2 67-97-0**, **Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(determination of **vitamins D2** and **D3** in pharmaceuticals by **supercrit.**-fluid extraction and HPLC with **UV** detection)

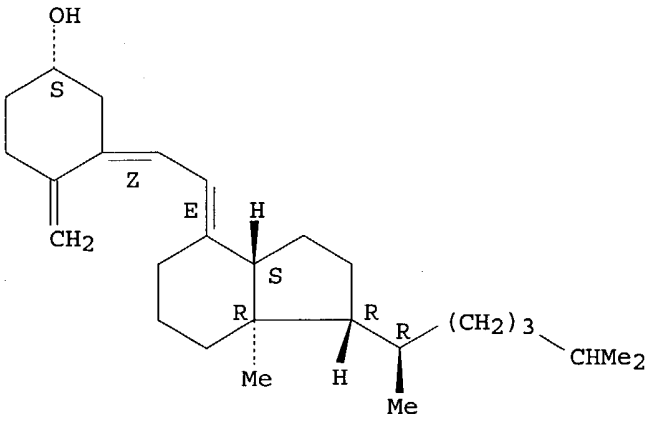
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 11 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2000:12675 HCAPLUS
DN 132:64451
ED Entered STN: 06 Jan 2000
TI Process for the preparation of **vitamin D3** and provitamin **D3**
IN **Johannsen, Monika**
PA F. **Hoffmann-La Roche** A.-G., Switz.
SO Eur. Pat. Appl., 7 pp.
CODEN: EPXXDW
DT Patent
LA German
IC ICM C07C401-00
CC 32-7 (Steroids)
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 969001	A2	20000105	EP 1999-111617	19990616 <--
	R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
	US 2001001801	A1	20010524	US 1999-335022	19990617 <--
	CN 1240209	A	20000105	CN 1999-108675	19990622 <--
	KR 2000006347	A	20000125	KR 1999-23466	19990622 <--
	JP 2000053640	A2	20000222	JP 1999-175755	19990622 <--
	BR 9903274	A	20000516	BR 1999-3274	19990622 <--
PRAI	EP 1998-111490	A	19980623 <--		

AB A process for obtaining **vitamin D3** or **previtamin D3** from a mixture of other steroids, such as dehydrocholesterol, lumisterol and tachysterol, is characterized by separating **vitamin D3** or **previtamin D3** by means of column **chromatog.** with **supercrit.** or liquid **carbon dioxide** as the mobile phase. Thus, **vitamin D3** was separated from a steroid mixture consisting of **vitamin D3**, **previtamin D3**, dehydrocholesterol, lumisterol and tachysterol, using a Hewlett-Packard **chromatograph** (HP G105A SFC) with **supercrit. CO2** (>>31° and >>7.3 MPa) as the mobile phase. Schematics for the separation procedure and a diagram of the **chromatog.** apparatus are presented.
ST column **chromatog** sepn **vitamin D3** **previtamin D3**
IT **Liquid chromatography**
Purification
Separation

Supercritical fluid chromatography
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **Silica gel, uses**
RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT Steroids, preparation
RL: PUR (Purification or recovery); REM (Removal or disposal); PREP (Preparation); PROC (Process)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **124-38-9, Carbon dioxide**, uses
RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

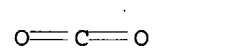
IT **67-97-0P, Vitamin D3 1173-13-3P, Previtamin D3**
RL: PUR (Purification or recovery); PREP (Preparation)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

IT **115-61-7**, Tachysterol 434-16-2, Dehydrocholesterol 474-69-1, Lumisterol
RL: REM (Removal or disposal); PROC (Process)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

RE.CNT 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Anon; EP 0969001 A2 HCAPLUS

IT **124-38-9, Carbon dioxide**, uses
RL: NUU (Other use, unclassified); USES (Uses)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

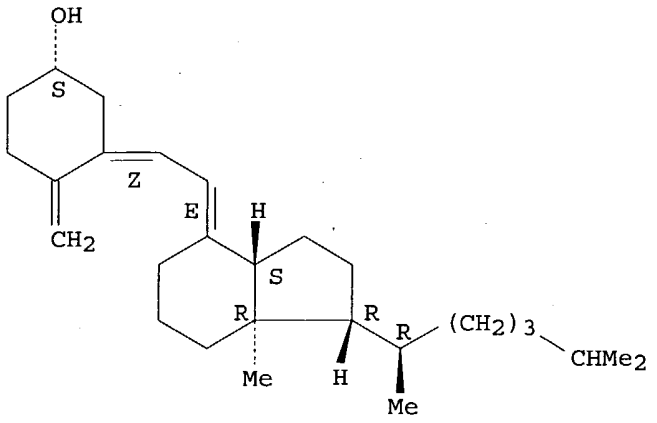
RN 124-38-9 HCAPLUS
CN Carbon dioxide (8CI, 9CI) (CA INDEX NAME)



IT **67-97-0P, Vitamin D3 1173-13-3P, Previtamin D3**
RL: PUR (Purification or recovery); PREP (Preparation)
(preparation of **vitamin D3** and provitamin **D3**
via separation from a mixture via column **chromatog.** with
supercrit. or liquid **carbon dioxide**)

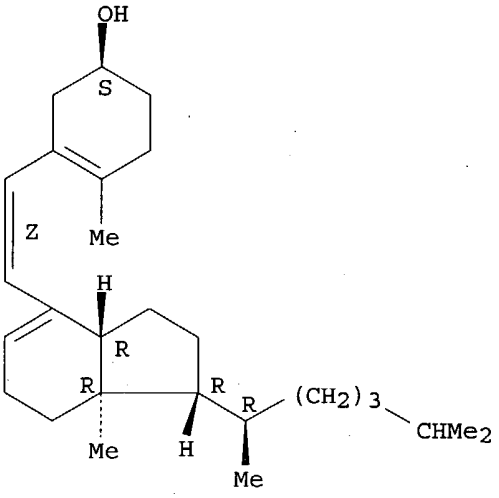
RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



RN 1173-13-3 HCAPLUS
CN 9,10-Secocholesta-5(10),6,8-trien-3-ol, (3β,6Z)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



IT **115-61-7**, Tachysterol
RL: REM (Removal or disposal); PROC (Process)
(preparation of **vitamin D3** and provitamin **D3** via separation from a mixture via column **chromatog.** with **supercrit.** or liquid **carbon dioxide**)
RN 115-61-7 HCAPLUS
CN 9,10-Secoergosta-5(10),6,8,22-tetraen-3-ol, (3β,6E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.

undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT Lipids, processes
RL: REM (Removal or disposal); PROC (Process)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT Breakfast cereal
(musli; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT Lipids, biological studies
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(nonpolar; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Fats and Glyceridic oils, biological studies
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(oat; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT Sterols
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(phyto-; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT Fatty acids, biological studies
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd., n-3; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Fatty acids, biological studies
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd., omega-6; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

IT Fatty acids, biological studies
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(polyunsatd.; method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2** and by introducing desired lipophilic substances using compressed **CO2**)

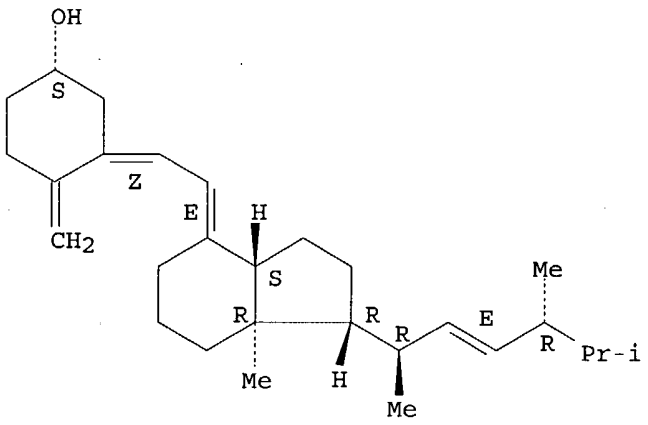
IT 58-95-7, Tocopherol acetate 59-02-9, α-Tocopherol 64-17-5D, Ethanol, polyunsatd. fatty acid esters, biological studies 463-40-1D, Linolenic acid, esters 506-32-1D, Arachidonic acid, esters **1406-16-2, Vitamin D** 1406-18-4, Vitamin E 6217-54-5, Cervonic acid 10417-94-4, Timnodonic acid 11103-57-4, Provitamin A 57828-26-9D, Lipoic acid, derivs.
RL: FFD (Food or feed use); BIOL (Biological study); USES (Uses)
(method for producing foodstuffs and/or feedstuffs by removing undesired lipid constituents using **supercrit. CO2**
and by introducing desired lipophilic substances using compressed **CO2**)

IT 74-98-6, Propane, biological studies
RL: FFD (Food or feed use); PEP (Physical, engineering or chemical

O=C=O

L79 ANSWER 22 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1961:49155 HCAPLUS
DN 55:49155
OREF 55:9533f-h
ED Entered STN: 22 Apr 2001
TI Evaluation of **ultraviolet irradiation** by determining the photochemical conversion of ergosterol to **vitamin D2**
AU Jerzykowski, Tadeusz
CS Inst. Med. Pracy, Zabrze, Pol.
SO Medycyna Pracy (**1960**), 11, 331-6
CODEN: MEPAAX; ISSN: 0465-5893
DT Journal
LA Unavailable
CC 11B (Biological Chemistry: Methods)
AB A method was developed for relative evaluation of the antirachitic activity of **ultraviolet-radiation** sources. A 0.1% ergosterol (I) solution in C6H6 under **CO2** or N is exposed under standardized time and area conditions to the **ultraviolet** light to be evaluated. A 0.5-ml. sample of the **irradiated** solution is treated with 0.5 ml. 1.4% citraconic anhydride in C6H6, left 30 min. in darkness, and evaporated in vacuo to dryness, the residue treated with 3 ml. CHCl3 and 2 ml. 1% AcCl in α-dichlorohydrin, let stand for 25 min., and extinction determined photometrically in a 5-cm. thick layer (Eb). Similar detns. are run with 0.1% solution of **nonirradiated** I (Ee) and 0.02% solution of crystalline **vitamin D2** (Ev). The % conversion = 100 (Eb - Ee)/(5Ev - Ee).
IT Electrophoresis, **Electrochromatography**
(immuno-)
IT **Ultraviolet** light
(**irradiation** by, evaluation of)
IT 57-87-4, Ergosterol
(ergocalciferol formation from, in **ultraviolet irradiation** evaluation)
IT **50-14-6**, Ergocalciferol
(ergosterol conversion to, in **ultraviolet irradiation** evaluation)
IT 66-02-4, Tyrosine, 3,5-diiodo-
(formation by thyroid and its determination, Cd effect on)
IT **50-14-6**, Ergocalciferol
(ergosterol conversion to, in **ultraviolet irradiation** evaluation)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 23 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1957:77001 HCAPLUS
DN 51:77001
OREF 51:13891g-i,13892a-i,13893a-c
ED Entered STN: 22 Apr 2001
TI The **vitamin D** series. XVIII. Partial synthesis of 5,6-trans-**vitamin D2**
AU Inhoffen, Hans H.; Kath, Joachim F.; Sticherling, Wolfgang; Bruckner, Klaus
CS Tech. Hochschule, Braunschweig, Germany
SO Ann. (**1957**), 603, 25-36
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 51, 8121f. All expts. were made under pure N. M.ps. are uncor. Neutral Number 2 Al2O3 was used in all **chromatograms**, and prepared either by the Woelm (W.) or the Brockmann (B.) method. Full details are given for all **chromatographic** sepns. The "Abbau" aldehyde, C21H34O, m. 56.5-58°, λ 238 mμ (ε 20,000) (cf. Windaus and Riemann, C.A. 37, 59768), (15 g.) in 400 cc. absolute EtOH, 105 g. 4-acetoxycyclohexanone, and 400 cc. 2% EtONa kept 2.5 hrs. at -20°, poured into H2O, extracted with Et2O, and the extract washed, dried, and evaporated gave a brown oil, freed from hydroxycyclohexanone and impurities by **chromatography** on Al2O3 (W.) and elution with C6H6, then with CH2Cl2, yielding 15.3 g. C27 ketone (I, R = H, R' = O) (Ia), which was acetylated with 168 cc. pyridine and 84 cc. Ac2O, added to N-saturated H2O, and extracted with Et2O giving I (R = Ac, R' = O) (Ib), isolated by adsorption on Al2O3 (W.) and eluted with C6H6 (either with or without petr. ether). On heating impure Ib at 90-100°/5 + 10-3 mm., 4-acetoxycyclohexanone, identified by its infrared (I.R.) spectrum, distilled The dry still residue, Ib, yellow oil, decomposed partially on attempted distillation at 210° in a high vacuum. Ib, dried but not distilled, λ 301, ε 24,600, **rechromatographed** and saponified at 20°, gave Ia, m. 135° (from aqueous MeOH), λ 300-302 ε 19,800. Another sample of Ib somewhat similarly purified and saponified gave Ia, m. 138°, λ 300, ε 21,300; the mixed m.ps. showed no depression. Ph3P:CH2 (from 4.1 g. Pn3PMeBr and 1.03 g. LiPh) treated in Et2O at 20° with constant stirring with 3.9 g. Ib in 500 cc. absolute Et2O, then refluxed 3 hrs., kept overnight, treated with N-saturated H2O, the Et2O phase removed, the mixture reextd. with Et2O, and all the exts. washed, dried, and evaporated gave 4.5 g. brown oil which, **chromatographed** on Al2O3 (W.), yielded Ph2 (removed by petr.

ether-C6H6, 4:1), then 1.59 g. 5,6-**vitamin D2** acetate, I (R = Ac, R' = CH2) (Ic), nearly colorless oil, λ 272 mμ, ε 20,200, whose I.R. spectrum is given and discussed. Ic (prepared from another sample of Ib) showed λ 272-3 mμ, ε 19,850. Ic (1.6 g.) in 150 cc. Et2O saponified with 75 cc. 3% KOH in MeOH 22 hrs. at 20° gave I (R = H, R' = CH2) (Id), m. 125-6° (preliminary sintering) (from Me2CO at 0°), λ 272-3 mμ, ε 24,600, [α]D20 74.8 ± 1° (C6H6), whose I.R. spectrum in CHCl3 was determined Prepared at room temperature from p-PhN2C6H4COCl and Id, I (R = p-PhN2C6H4CO, R' = CH2) (Ie) formed red crystals, m. 114-17° (preliminary sintering) (from Me2CO), λ 276-8 and 322 mμ (ε 30,000 and 29,700, resp.), [α]D20 25.5 ± 1.5° (CHCl3), whose I.R. spectrum is discussed. All spectra are in harmony with the structure assigned to Id, 0.95 g. of which in 74 cc. PhMe and 14 cc. cyclohexanone concentrated to about 60 cc. to insure drying, cooled, and refluxed 45 min. with 0.6 g. (iso-PrO)3Al, poured into H2O saturated with N, extracted with Et2O, and the exts. washed to neutrality, dried, evaporated, and heated 1 hr. at 90°/5.10-3 mm. gave as still residue 853 mg. crude **vitamin D2** ketone (II), yellow oil, λ 263-4 mμ ε 11,100 (Et2O), 266 mμ, ε 10,000 (MeOH); its I.R. spectrum shows the characteristic bands of a dieneone. The semicarbazone of II, m. 209° (from CHCl3-MeOH), decomposed readily by air and light, λ 292 mμ, ε 29,350, [α]D20 24 ± 1° (CHCl3), is identical with that isolated by Trippett (C.A. 49, 6286d) from cis-**vitamin D2.** Ia (3.5 g.) in 30 cc. dihydropyran with a drop of POCl3 kept 2 hrs. at room temperature, shaken with saturated NaHCO3 solution, extracted repeatedly with Et2O, the extract dried, evaporated, taken up 3 times in absolute C6H6, and each time evaporated to dryness in vacuo, heated at 80°/10-3 mm. to remove impurities, the still residue (4.61 g.) in 30 cc. C6H6 filtered through 80 g. Al2O3 (B.), and the eluate evaporated gave 4.2 g. stable tetrahydropyranyl ether (III) of Ia (not analyzed), whose I.R. spectrum showed no OH band, λ 302 mμ, ε 22,400. Ia (1.5 g.) in CH2:CHOEt heated 18 hrs. at 125-35° in a sealed tube, **chromatographed** on Al2O3 (B.) with petr. ether and petr. ether-C6H6 as eluants gave 1.168 g. EtOCHMe ether (IV) of Ia (not analyzed), λ 302 mμ, ε 25,000 and 26,800, whose I.R. spectrum indicated etherification. Crystalline **vitamin D2** (V) (source not given) subjected to similar slightly modified etherifications and purifications through Al2O3 (B.) gave the following ethers of V: 70% EtOCHMe (VI), colorless oil (not analyzed), λ 266 mμ, ε 16,300, and 75% tetrahydropyranyl (VII), C33H52O2, yellow oil, λ 266 mμ, ε 13,200. III (1.31 g.) with Ph3P:CH2 (from 0.97 g. Ph3PMeBr) in absolute Et2O warmed 90 min. at 60°, kept 15 hrs. at 20°, filtered, washed with Et2O, and the combined Et2O exts. washed, dried, evaporated, and **chromatographed** on Al2O3 (B.) gave, after removal of Ph2 and elution with petr. ether-C6H6, 28% tetrahydropyranyl ether of Id, colorless oil, λ 272 mμ, ε 21,200 or 2,300, whose I.R. spectrum showed the absence of a C:O but the presence of a :CH2 band. Similarly, IV gave 34% EtOCHMe ether of Id, colorless, λ 272 mμ, ε 22,700. IV (0.1 g.) in 3 cc. dioxane refluxed 3 hrs. with 0.6 cc. 1% aqueous (CO2H)2, cooled, poured into 100 cc. H2O, extracted with Et2O, the exts. washed with aqueous NaHCO3 and H2O, dried, evaporated, and the residue taken up in C6H6 and **chromatographed** gave 62 mg. Ia. VI, decomposed under varying conditions with (CO2H)2, usually gave mixts. of the original ether and V. In 1 experiment 1.6 g. VI in 25 cc. dioxane refluxed 3 hrs. with 10 cc. 1% (CO2H)2 gave 1.43 g. oil, which **chromatographed** in petr. ether on Al2O3 (B.) and the products eluted with C6H6 and then with

Et2O, yielded 735 mg. isovitamin D2, pale yellow oil, λ 276 and 286 mμ, highest ε 25,200 and 27,200, resp., and 334 mg. V, glassy solid, λ 266 mμ, highest ε 18,600. VI in petr. ether shaken with 1% aqueous NaHSO3 was recovered unchanged. VI (1.3 g.) in 25 cc. absolute EtOH with small amts. of Hg(OAc)2 heated 18 hrs. in a bomb tube and **chromatographed** gave 0.8 g. V, glass, identified by its I.R. spectrum. VII (0.3 g.) in 15 cc. dioxane treated 18 hrs. at 20° with H2O (distilled over KOH) and saturated with N and with a gentle stream of **CO2** was recovered unchanged. The tetrahydropyranyl ether of Id (0.16 g.) in 80 cc. warm dioxane refluxed 0.5 hr. with 0.7 cc. 8% (CO2H)2 gave a yellow oil, which, even after **chromatography** on Al2O3, yielded fractions that were mixts. The EtOCHMe ether of Id (0.45 g.) in 12 cc. dioxane and 2.5 cc. 1% aqueous (CO2H)2 kept 130 min. at 90° and **chromatographed** gave 39% iso-**vitamin D2**, colorless crystals, λ 287-8 mμ, ε ranging from 9900 to 43,000, identified by its **ultraviolet** and I.R. spectra. Id showed only 0.8% of the antirachitic activity manifested by cis-**vitamin D2**. When Id was **irradiated** with **ultraviolet** light, its activity increased 6-fold. This, however, is not unequivocal evidence that a partial synthesis of cis-**vitamin D2** had occurred.

IT Rickets
(antirachitic substances, 5,6-trans-**vitamin D** and related compds. as)

IT Infrared spectra
(of **vitamin D** and related compds.)

IT Benzoic acid, p-phenylazo-, esters with trans-**vitamin D2**
Cyclohexanone, 4-(1-ethoxyethoxy)-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
Indan, 4-{2-[5-(1-ethoxyethoxy)-2-methyl-enecyclohexylidene]ethylidene}hexahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-

IT Pyran, tetrahydro-2-{4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethyl}cyclohexyloxy}-
(stereoisomers)

IT Acetaldehyde, vinyl acetal polymers
Acetaldehyde, vinyl acetal polymers
(**vitamin D2**-related acetals)

IT **1406-16-2**, **Vitamin D**
(-related compds.)

IT 474-63-5, Ergosta-5,24(28)-dien-3β-ol
(and esters)

IT 41043-88-3, Cyclohexanone, 4-hydroxy-, acetate
(formation from 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}cyclohexanone)

IT **50-14-6**, Ergocalciferol
(isomers of, and derivs.)

IT **51744-67-3**, Isovitamin, D2 **104398-83-6**, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-, semicarbazone **108749-29-7**, Cyclohexanone, 2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-4-(tetrahydropyran-2-yloxy)-
115918-16-6, Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-, acetate
117342-65-1, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-
(preparation of)

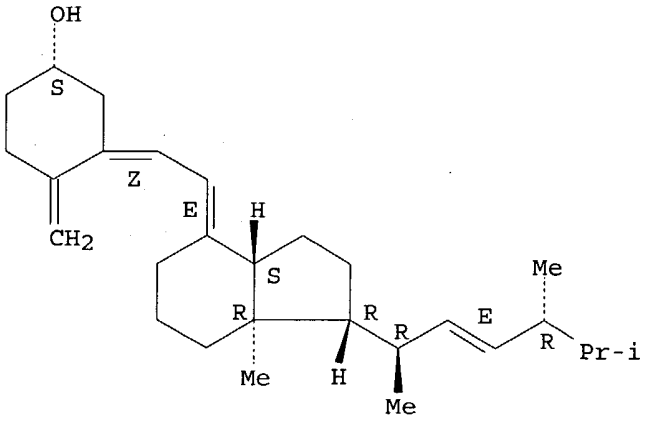
IT **50-14-6**, Cyclohexanol, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
(stereoisomers, and derivs.)

IT **1406-16-2**, **Vitamin D**
(-related compds.)

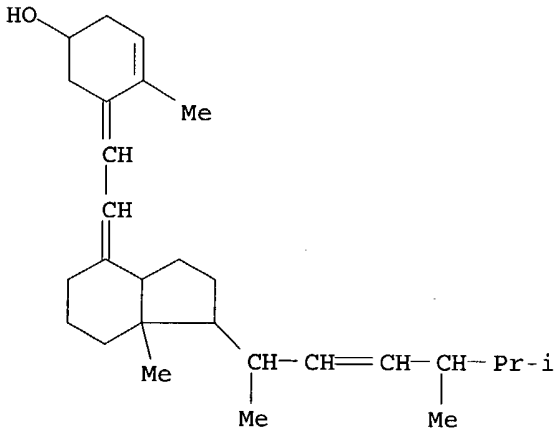
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
IT **50-14-6**, Ergocalciferol
(isomers of, and derivs.)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI)
(CA INDEX NAME)

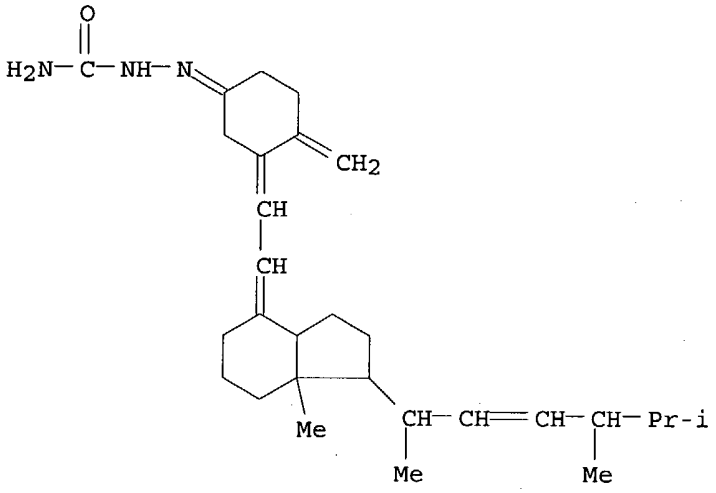
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



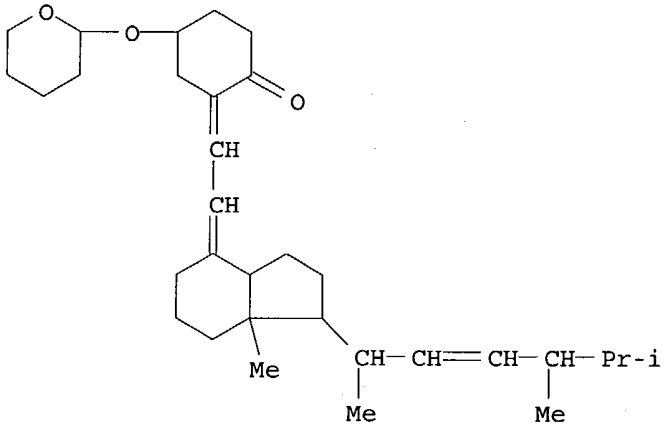
IT **51744-67-3**, Isovitamin, D2 **104398-83-6**, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-, semicarbazone **108749-29-7**, Cyclohexanone, 2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-4-(tetrahydropyran-2-yloxy)- **115918-16-6**, Cyclohexanone, 4-hydroxy-2-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-, acetate **117342-65-1**, Cyclohexanone, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-(3aH)-indanylidene]ethylidene}-
(preparation of)
RN 51744-67-3 HCAPLUS
CN 9,10-Secoergosta-1(10),5,7,22-tetraen-3-ol, (3β,5E,7E,22E)- (9CI)
(CA INDEX NAME)



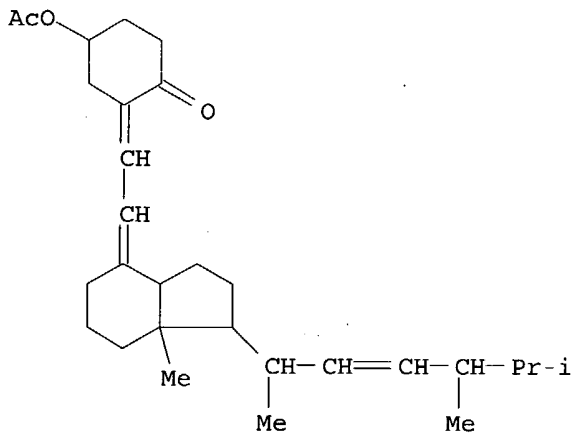
RN 104398-83-6 HCAPLUS
CN Cyclohexanone, 4-methylene-3-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-, semicarbazone (6CI) (CA INDEX NAME)



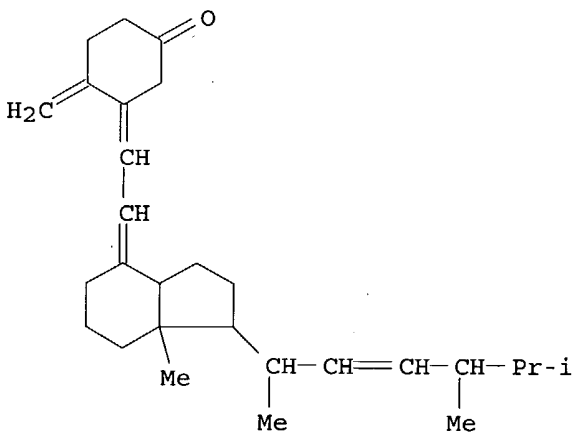
RN 108749-29-7 HCAPLUS
CN Cyclohexanone, 2-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-4-(tetrahydropyran-2-yloxy)- (6CI) (CA INDEX NAME)



RN 115918-16-6 HCAPLUS
CN Cyclohexanone, 4-hydroxy-2-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]-, acetate (6CI) (CA INDEX NAME)

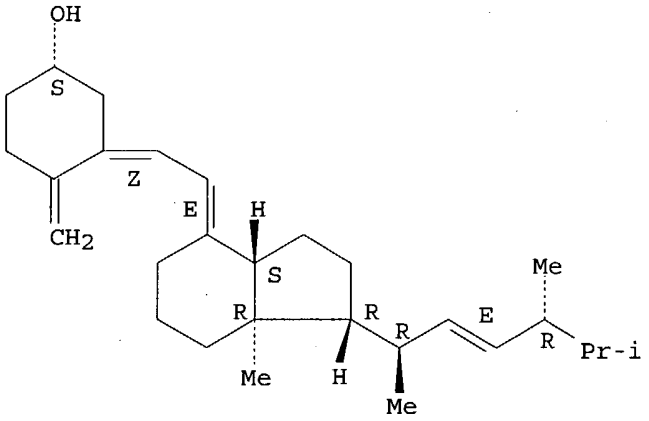


RN 117342-65-1 HCAPLUS
CN Cyclohexanone, 4-methylene-3-[2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene]- (6CI) (CA INDEX NAME)



IT **50-14-6**, Cyclohexanol, 4-methylene-3-{2-[tetrahydro-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4(3aH)-indanylidene]ethylidene}-
(stereoisomers, and derivs.)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 24 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1956:89264 HCAPLUS
DN 50:89264
OREF 50:16808i,16809a-i,16810a-b
ED Entered STN: 22 Apr 2001
TI **Vitamin D** series. IX. Ring A precursors for syntheses of tachysterol and its analogs
AU Inhoffen, Hans Herloff; Weissermel, Klaus; Quinkert, Gerhard
CS Tech. Hochschule, Braunschweig, Germany
SO Chemische Berichte (**1955**), 88, 1313-21
CODEN: CHBEAM; ISSN: 0009-2940
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
OS CASREACT 50:89264
AB cf. C.A. 50, 380e, 8573a. A solution of 4.53 g. 1-methyl-cis-1,2-cyclohexanediol (prepared from 1-methylcyclohexene with KMnO4) in 5 cc. CCl4, 8 cc. CHCl3, and 4.4 cc. absolute pyridine treated dropwise below 5° with 3.26 g. tert-BuOCl, kept overnight, extracted with Et2O, and the Et2O residue (4.2 g.) in petr. ether **chromatographed** on Al2O3 and eluted with petr. ether, then with Et2O and Et2O-MeOH (10:1) gave, resp., 0.45 g. tert-BuOH and 1.9 g. 1-hydroxy-1-methyl-2-cyclohexanone (I) (semicarbazone, m. 200-1°); the column treated with dilute H2SO4 and extracted with CHCl3 gave δ-acetylvaleric acid (semicarbazone, needles from EtOH, m. 143.5°). A stirred solution of 70 g. freshly prepared moist 2-hydroxycyclohexanone in 700 cc. hot H2O treated with concentrated HCl, then during 5-10 min. with 296 g. FeCl3.-6H2O in 150 cc. H2O, cooled to 40°, treated with 220 g. (NH4)2SO4, and extracted with CH2Cl2 gave 1,2-cyclohexanedione, b23 81-5°, m. 38-40°, which gave I with MeMgBr. A solution of 3 g. Li in 800 cc. liquid NH3 treated with dry C2H2 until colorless, then during, and for 2 hrs. after the dropwise addition of 20 g. I in 50 cc. absolute Et2O, kept 3 hrs., treated with 250 cc. Et2O and dried NH4Cl, and evaporated slowly gave 13 g. 1-methyl-2-ethynyl-1,2-cyclohexanediol, b2.5 58-96°. A solution of 78 g. 4-acetoxycyclohexanone in 500 cc. Et2O treated dropwise at -5° with 900 cc. MeMgBr solution (from 18 g. Mg), kept at 0° 10 hrs., decomposed (chilling) with the calculated amount dilute H2SO4, saturated with NaCl, and extracted with Et2O gave 34.5 g. 4-acetoxy-1-methylcyclohexanol (II), b0.4 71-3°; 18 g. KHSO4 and 30 g. II heated at 90°/0.5 mm., the product distilled with N, and the distillate extracted with Et2O and neutralized gave 12.3 g. 4-acetoxy-1-methylcyclohexene (III), b23 90°. Li (11

g.) in about 1.3 l. liquid NH3 treated dropwise with stirring with 40 g. p-MeC6H4OMe in 60 cc. Et2O, then after 15 min. with EtOH until colorless, then after evaporating most of the NH3, with 300 cc. Et2O and a saturated solution of 95 g. NH4Cl, the stirring was continued until nearly all the NH3 was removed, the aqueous solution extracted with addnl. Et2O, the combined Et2O solns. washed with saturated NH4Cl, concentrated to 250 cc., stirred 90 min. with 42 g. (CO2H)2 in 550 cc. H2O, the aqueous layer saturated with NaCl and extracted with addnl. Et2O, and the combined Et2O solns. neutralized, dried, and evaporated gave 23.6 g. 1-methyl-1-cyclohexen-4-one [4-methyl-3-cyclohexen-1-one] (IV), b29 75-84° [semicarbazone, m. 172°; 2,4-dinitrophenylhydrazone, m. 130° (both from EtOH)]. IV (101 g.) in 200 cc. Et2O added dropwise under N to 11 g. LiAlH4 in 500 cc. Et2O, the mixture refluxed 1 hr., decomposed in an ice-salt bath with saturated NH4Cl solution, and the solid extracted twice with 400 cc. total addnl. Et2O gave 95.5 g. 1-methyl-1-cyclohexen-4-ol (V), b30-1 90-6° [3,5-dinitrobenzoate, m. 113-13.5° (from EtOH); phenylurethan, m. 108.5-9.5° (from C6H6)]. V (39 g.), 58.8 g. freshly-distilled dihydropyran, and a drop POCl3 shaken with cooling until warm, kept 24 hrs., heated on a water-bath 20 min., cooled, diluted with Et2O, the solution added to 20% KOH, and the Et2O residue distilled gave 65.1 g. 4-(2-tetrahydropyranyloxy)-1-methyl-1-cyclohexene (VI), b2.5-3 90-1.5°. Treating 250 cc. 15% NaOH at -10° with 105 cc. chilled 30% H2O2, then portionwise (stirring) with 75 g. finely powdered o-C6H4-(**CO2**)2O, then with 250 cc. chilled 20% H2SO4, extracting the filtrate with Et2O, washing the exts. with 40% (NH4)2SO4, and drying with Na2SO4 gave a perphthalic acid solution (VII). III in 1 volume absolute Et2O added slowly to an equimolar amount VII, kept 2 days in an icebox, 3 days at room temperature, stirred into chilled aqueous NaOH, the aqueous layer extracted with Et2O, the combined Et2O solns. treated with FeSO4 solution to a neg. KI test, the aqueous layer extracted with Et2O, and the combined Et2O solns. washed with a little H2O, dried (Na2SO4), and evaporated gave 62% 4-acetoxy-1-methyl-1,2-oxidocyclohexane (VIII), b24 113-19°. VI gave 58% 4-(2-tetrahydropyranyloxy)-1-methyl-1,2-oxidocyclohexane, b5 116-19°. Refluxing 30 g. VIII with 11 g. KOH in 100 cc. MeOH 30 min., diluting with H2O, saturating with NaCl, extracting with Et2O, then during 3 days with CHCl3, and evaporating the organic solns. gave 11.6 g. 4-hydroxy-1-methyl-1,2-oxidocyclohexane, b26 126-6.5°. Heating 14 g. VIII and 140 cc. 0.05N HCl on a water-bath 15 min., saturating with NaCl, and extracting 3 days with CHCl3 gave 12.5 g. 4-acetoxy-1-methyl-trans-1,2-cyclohexanediol, b0.02 126-32°, m. 146-6.5° (from MeOH-hexane). A stirred solution of 39.2 g. VI in 400 cc. EtOH treated at -35° (finally at -15°) with 26 g. KMnO4 and 29 g. crystalline MgSO4 in 520 cc. H2O during 1.5 hrs., filtered from the MnO2, concentrated in vacuo, saturated with NaCl, extracted with CHCl3, and the exts. dried and evaporated gave 14.3 g. 4-(2-tetrahydropyranyloxy)-1-methyl-cis-1,2-cyclohexanediol, b0.005 124-37°, m. 101.5°. A solution of 30 g. 1-methyl-2-ethynylcyclohexene (prepared from 1-methyl-2-ethynyl-2-cyclohexanol with POCl3-pyridine) in 75 cc. absolute Et2O treated dropwise under N with 520 cc. LiMe solution (1.515 g. active Li), refluxed 30 min., cooled, refluxed 30 min. with 11.1 g. I in 30 cc. Et2O, cooled, decomposed with chilled saturated NH4Cl. solution, the crude product (20.6 g., λ 229-30 mμ) in C6H6 **chromatographed** on Al2O3, eluted with C6H6, C6H6-Et2O (2:1), Et2O-C6H6 (3:1), Et2O, and Et2O-MeOH (up to 7%), and the eluates (except with C6H6) evaporated gave 7.2 g. α-(1,2-

dihydroxy-2-methylcyclohexenyl)-β-(2-methyl-1-cyclohexenyl)-acetylene (IX), b0.01 130-6°, λ 229-30 mμ (ε 13250, MeOH); 1.2 g. IX in 10 cc. absolute Et2O treated dropwise with 1.6 g. P2I4 in 12 cc. CS2, poured after 10 min. on aqueous NaOH, ice, and Et2O, shaken with aqueous Na2S2O3, the organic solution evaporated, and the residue (410 mg.) in petr. ether filtered through Al2O3 gave O-sensitive α,β-bis(2-methyl-1-cyclohexenyl)acetylene, λ 270-2 mμ (ε 12280, MeOH).

IT **Chromatography and Adsorption analysis**
(of cyclohexane derivs. (unsatd.))

IT 1,2,4-Cyclohexanetriol, 1-methyl-, trans-, 4-acetate
1,2-Cyclohexanediol, 1-ethynyl-2-methyl-
1,2-Cyclohexanediol, 1-methyl-2-(2-methyl-1-cyclohexen-1-ylethynyl)-
1,2-Cyclohexanediol, 1-methyl-4-(tetrahydropyran-2-yloxy)-, cis-
7-Oxabicyclo[4.1.0]heptan-3-ol, 6-methyl-
7-Oxabicyclo[4.1.0]heptane, 1-methyl-4-(tetrahydropyran-2-yloxy)-
Pyran, 2-(3,4-dihydroxy-4-methylcyclohexyloxy)tetrahydro-
Pyran, tetrahydro-2-(6-methyl-7-oxabicyclo[4.1.0]hept-3-yloxy)-

IT **1406-16-2, Vitamin D**
(-related compds.)

IT 5259-65-4, 3-Cyclohexen-1-one, 4-methyl- 51422-70-9, 3-Cyclohexen-1-ol, 4-methyl-
(and derivs.)

IT **115-61-7**, Tachysterol
(and related compds.)

IT 765-87-7, 1,2-Cyclohexanedione 3476-78-6, Cyclohexanone, 2-hydroxy-2-methyl- 17713-68-7, Heptanoic acid, 6-oxo-, semicarbazone 32591-08-5, Cyclohexene, 1-ethynyl-2-methyl- 52718-65-7, 1,2-Cyclohexanediol, 1-methyl-, cis- 98553-41-4, Cyclohexanone, 2-hydroxy-2-methyl-, semicarbazone 100227-28-9, 7-Oxabicyclo[4.1.0]heptan-3-ol, 6-methyl-, acetate 100315-12-6, Pyran, tetrahydro-2-(4-methyl-3-cyclohexen-1-yloxy)- 131974-95-3, Acetylene, bis(2-methyl-1-cyclohexen-1-yl)- 479075-85-9, 1,4-Cyclohexanediol, 1-methyl-, 4-acetate
(preparation of)

IT **1406-16-2, Vitamin D**
(-related compds.)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

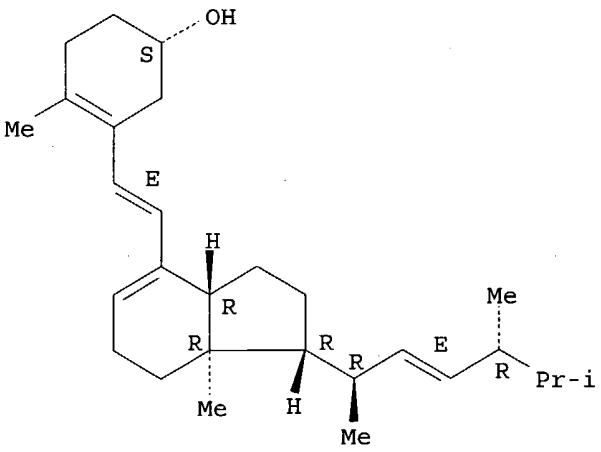
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

IT **115-61-7**, Tachysterol
(and related compds.)

RN 115-61-7 HCAPLUS

CN 9,10-Secoergosta-5(10),6,8,22-tetraen-3-ol, (3β,6E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 25 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1956:57446 HCAPLUS
DN 50:57446
OREF 50:10848b-f
ED Entered STN: 22 Apr 2001
TI Methods for the determinations of fat-soluble **vitamins** A, D2, **D3**, and E
AU Vendt, V. P.
CS Biochem. Inst., Acad. Sci. Ukr. S.S.R., Kiev
SO Vitaminy, Akad. Nauk Ukr. S.S.R. (**1953**) 7-29
DT Journal
LA Unavailable
CC 11B (Biological Chemistry: Methods and Apparatus)
AB Detns. of fat-soluble **vitamins** A, D2, **D3**, and E are thoroughly discussed and the following methods are considered the most suitable. **Vitamin** A: colorimetric reaction with 1,3-dichloropropan-2-ol (C.A. 41, 2536g) activated by 1-2% HCl added to the reagent; colorimetric, photocolorimetric, and spectrophotometric methods are described. **Vitamin** A as well as carotene can be determined in the same sample by using wave lengths of 550 and 750 mμ, resp. **Vitamin D2** (calciferol): interference of other sterols is eliminated by digitonin treatment of samples (0.5 ml. 2% aqueous suspension of digitonin per 7 ml. of the solution made up from 25 g. $SbCl_5$ dissolved in 100 ml. dichloroethane), removal of water by addition of anhydrous $MgSO_4$ or $NaSO_4$ to the mixture, filtration, and color formation with the $SbCl_5$ reagent; the colorimetric or spectrophotometric measurements of the color formed is made (within 5-10 min. after the addition of the $SbCl_5$ reagent) at 430 and 530 mμ, and the extinction coefficient of **vitamin D2** (ED2) is then calculated by ED2 = 1.23 E430 - 0.62 E530. The measurements of the two wave lengths is essential since by **irradiating** ergosterol and **chromatographing** (through activated Al_2O_3) the reaction mixture, 4 substances have been isolated which gave the color reaction with the $SbCl_5$ reagent. However, only one addnl. measurement at 530 mμ is required to get the quant. determination of **vitamin D2** in the samples. **Vitamin D3** is determined by a method similar to that of **vitamin D2**. **Vitamin** E: color formation with HNO_3 and the photocolorimetric measurement of the color formed at 470 mμ. To remove various interfering substances from the exptl. samples containing **vitamin** E, **chromatography** (Al_2O_3) is used, thus giving the following steps for the determination: sampling, saponification of the sample (butter), extraction of the unsapond. fraction by Et_2O, evaporation of Et_2O, solution of the residue in

dichloroethane, **chromatographic** filtration of the solution under a **CO2** stream, evaporation of dichloroethane, solution of the residue in EtOH, color formation with HNO3, and spectrophotometric determination of the color formed.

IT Carotene
(determination of)

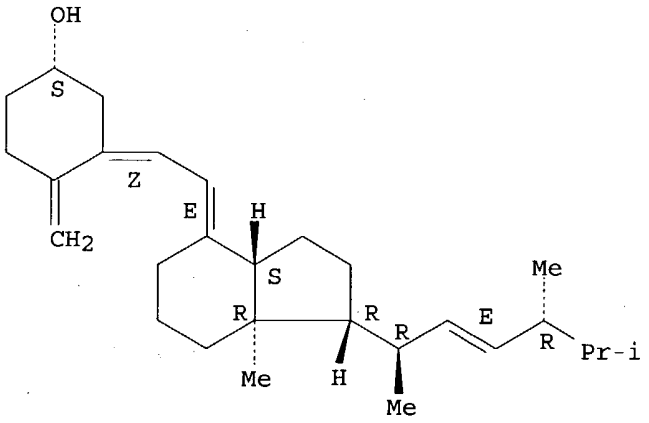
IT **50-14-6**, **Vitamin**, **D2 67-97-0**, **Vitamin**, **D3** 1406-18-4, Vitamin E 11103-57-4, Vitamin, A
(determination or assay of)

IT **50-14-6**, **Vitamin**, **D2 67-97-0**, **Vitamin**, **D3**
(determination or assay of)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

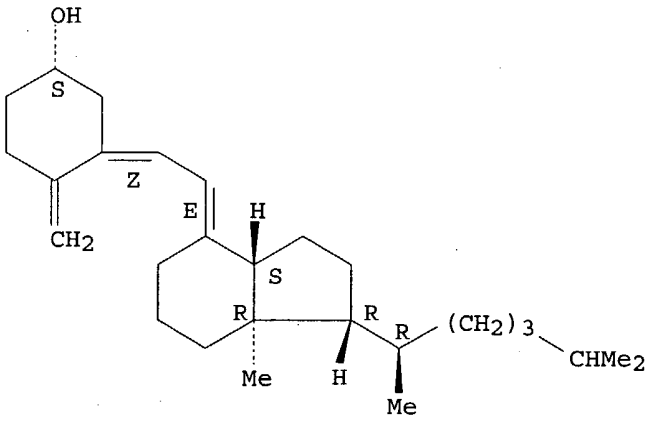
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L79 ANSWER 26 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1955:49277 HCAPLUS
DN 49:49277
OREF 49:9526b-i,9527a-c
ED Entered STN: 22 Apr 2001
TI The **vitamin D** series. III. Synthesis of a hydrindan derivative by the diene synthesis
AU Inhoffen, Hans Herloff; Kramer, Hans
CS Tech. Hochschule, Braunschweig, Germany
SO Chemische Berichte (**1954**), 87, 488-96
CODEN: CHBEAM; ISSN: 0009-2940
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
OS CASREACT 49:49277
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 49, 6291c. The diene reaction between AcOCH:CHCH:CH2 (I) and 1-methyl-1-cyclopentene-4,5-dione (II) produces an hydrindan derivative in which the AcO and Me groups are ortho to each other. To a solution of 120 g. 2-chlorocyclopentanone in 1200 cc. H2O heated to 100° 500 g. FeCl3.6H2O in 250 cc. H2O was added dropwise with stirring, the solution cooled to 40°, 360 g. (NH4)2SO4 added, the solution cooled and extracted with Et2O or CH2Cl2 from which 80.5 g. 1,2-cyclopentanedione (III), b16 87-8°, m. 55-6°, was obtained. III (60 g.) in 250 cc. Et2O was added to a MeMgBr solution containing 45 g. Mg in about 600 cc. Et2O, the mixture refluxed 20 min., decomposed with H2O and dilute H2SO4, continuously extracted with Et2O for 2 hrs., and the extract fractionated; material up to b17 78° was removed, and 24.8 g. 1-methyl-1-cyclopenten-5-one (IV), b. 157-8°, distilled at 760 mm.; in some cases, some 1-methylcyclopentan-1-ol-2-one, b17 79-81°, was obtained, which is dehydrated to IV with KHSO4. The SeO2 oxidation product of IV from four 6-g. runs, combined after removal of Se, freed from HOAc at 20 mm., distilled at 12 mm., and recrystd. from Et2O and petr. ether gave 44% II, m. 84.5°. II (2 g.) in 50 cc. C6H6, 7 cc. pure I, and 15 mg. methylene blue, heated in a bomb 40 hrs. at 120° and filtered, precipitated almost immediately 2.2 g. (55%) 7-acetoxy-8-methyl-5-hydrindene-1,2-dione (V), m. 123.5° (from cyclohexane or Et2O-petr. ether), λmaximum 259 mμ (ε 6100, MeOH) and 294 mμ (N/200 NaOH), violet with FeCl3. V (36 mg.) in 20 cc. C6H6 was boiled 2 hrs. with 75 mg. o-C6H4(NH2)2 in 10 cc. EtOH and 0.5 cc. HOAc, the solvent evaporated in vacuo, impurities removed by precipitation from Et2O solution with petr. ether, the solvents evaporated, and the residue in 1:1 Et2O-petr. ether **chromatographed** on Al2O3; elution with 80% C6H6 in petr. ether separated the quinoxaline derivative of V, λmaximum 240 and 320 mμ (ε 24800 and 8750, resp.). V (50 mg.) in a little EtOH warmed briefly with several drops of a solution of 1 g. H2NCONHNH2.HCl and 1.5 g. NaOAc in 5 cc. H2O gave after 30 hrs. the disemicarbazone of V, m. 236-6.5°. II (2 g.) in 50 cc. C6H6, 5 cc. I, 2.5 cc. Ac2O, and 15 mg. methylene blue, treated as above, the C6H6 removed in vacuo, the residue taken up in Et2O, filtered over activated C, the solution concentrated, and the product crystallized from cyclohexane gave 2.9 g. 2-enol acetate (VI) of V, m. 73.5°, λmaximum 229-30 mμ, sometimes obtained as a polymorph, m. 78.5°. VI (100 mg.) in 1.5 cc. EtOH, warmed 30 min. with 1.2 cc. 0.75N K2CO3, acidified with dilute H2SO4, and extracted with Et2O, gave 60.5 mg. (72%) V. VI (2 g.) in 60 cc. absolute Et2O added dropwise to 1.6 g. LiAlH4 in 80 cc. Et2O, and boiled 1 hr., gave 1.30 g. (93.5%) 1 (or 2),7-dihydroxy-8-methyl-5-hydrinden-2(or 1)-one (VII), b10 125-30°, which gave a Ag mirror with Tollens reagent. LiAlH4 reduction of V gave VII. VII (50 mg.) refluxed 30 min. with 2 cc. Ac2O, poured into H2O, extracted with Et2O, and the material from evaporation of the extract warmed with H2NCONHNH2-NaOAc solution and let stand 24

hrs., gave the semicarbazone of VII diacetate, m. 217-18° (from EtOAc). Oxidation of 1.5 g. VII in 200 cc. H2O 48 hrs. at room temperature with 2.9 g. NaIO4.2H2O in 200 cc. H2O and 100 cc. 2N H2SO4, addition of 2N NaOH to bring the solution to pH 2-3, extraction with Et2O and washing with NaHCO3 and dilute H2SO4 under N, and crystallization from a small amount Et2O gave 1.1 g. (75%) crude cyclohemiacetal lactone, probably VIII, m. 100-5°, which was neutral, gave no semicarbazone, and was colorless with Schiff reagent; on titration VIII consumed 1 mole NaOH, and after treatment with NaHCO3 VIII gave a rose Schiff test. To a cold solution of 800 mg. VIII in 7.5 cc. Me2CO3.75 cc. of a solution containing 100 g. CrO3 and 160 g. concentrated H2SO4/500 cc. was added, the mixture shaken 15 min. at room temperature, 10 cc. H2O added, **CO2** evolved, the solution extracted with Et2O, and the extract distilled, giving 400 mg. 2-methyl-3-oxo-4-cyclopentene-1-acetic acid (IX), b1 120-30°, m. 98-9.5° after recrystn. from C6H6-petr. ether and Et2O-petr. ether, λmaximum 226 mμ (ε 9600). IX (70 mg.) in 7:3 EtOH-C6H6 containing several drops concentrated H2SO4 refluxed 4 hrs., part of solvent distilled slowly, the solution diluted with Et2O, washed with NaHCO3, and the Et2O solution evaporated and boiled 2 hrs. with aqueous alc. H2CONHNH2 solution gave the semicarbazone of IX Et ester, λmaximum 264 mμ (ε 16000).

IT Synthesis
(diene, of **vitamin D**-related compds.)

IT Spectra
(of **vitamin D**-related compds.)

IT 1-Indanone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-
1-Indanone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-, semicarbazone diacetate
11H-Indeno[1,2-b]quinoxalin-1-ol, 1,4,4a,11a-tetrahydro-4a-methyl-, acetate (ester)
11H-Indeno[1,2-b]quinoxalin-4-ol, 1,4,4a,11a-tetrahydro-4a-methyl-, acetate (ester)
2-Indanone, 3a,4,7,7a-tetrahydro-1,4(or 1,7)-dihydroxy-7a-methyl-, semicarbazone diacetate
3-Cyclohexene-1-acetic acid, 6-methyl-5-oxo-
3-Cyclohexene-1-acetic acid, 6-methyl-5-oxo-, ethyl ester semicarbazone
Indone, 3a,4,7,7a-tetrahydro-2,4(or 2,7)-dihydroxy-7a-methyl-, diacetate

IT 1,2-Indandione, 3a,4,7,7a-tetrahydro-4(or 7)hydroxy-7a-methyl-
(derivs.)

IT Bromine silver benzoate, (BzO)2AgBr
(reaction with aromatic compds.)

IT **1406-16-2, Vitamin D**
(-related compds.)

IT 5090-76-6, Cyclohexanepropionic acid, 2-carboxy-2-methyl-6-oxo-
103386-94-3, 1,4(3aH)-Indandione, tetrahydro-7a-methyl-
(and derivs.)

IT 496-10-6, Indan, hexahydro-
(derivs.)

IT 1120-73-6, 2-Cyclopenten-1-one, 2-methyl- 3008-40-0, 1,2-Cyclopentanedione 55767-59-4, Cyclopentanone, 2-hydroxy-2-methyl-
(preparation of)

IT 10130-95-7, 3-Cyclopentene-1,2-dione, 3-methyl-
(reaction with 1,3-butadienyl acetate)

IT 1515-76-0, 1,3-Butadien-1-ol, acetate
(reaction with 3-methyl-3-cyclopentene-1,2-dione)

IT **1406-16-2, Vitamin D**
(-related compds.)

RN 1406-16-2 HCAPLUS

CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 27 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1955:32479 HCAPLUS
DN 49:32479
OREF 49:6292g-i,6293a-g
ED Entered STN: 22 Apr 2001
TI The **vitamin D** series. II. Partial synthesis of the isotachysterol methyl ether
AU Inhoffen, Hans Herloff; Weissermel, Klaus
CS Tech. Hochschule, Braunschweig, Germany
SO Chemische Berichte (**1954**), 87, 187-93
CODEN: CHBEAM; ISSN: 0009-2940
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
OS CASREACT 49:32479
GI For diagram(s), see printed CA Issue.
AB To study the chemical and phys. properties of **vitamin D** the attempt is made to prepare other open-ring steroids with conjugated triene systems. Treating 154 g. 4-methoxycyclohexanone in 200 cc. ether with MeMgBr from 43.5 g. Mg gives 84% 4-methoxy-1-methylcyclohexanol, b19 106-7°, m. 21.5°, which (72 g.), heated 2 hrs. at 140° with 50 g. KHSO4, gives 82.5% 4-methoxy-1-methylcyclohexene (I), b80 91-2°. Treating 20 g. I in 80 cc. CCl4 with (CH2CO)2NBr a few min. at 60-70°, adding 10-20 cc. petr. ether, and keeping the mixture in a refrigerator gives 55% 6-bromo-4-methoxy-1-methylcyclohexene (II), b0.03 60-5°, which (105 g.) is heated in 100 cc. AcOH 2 hrs. at 130-50° with 80 g. anhydrous KOAc, the mixture diluted with H2O, extracted with ether, and the residue of the ether extract fractionally distilled, giving 3 fractions: (a) 25 g., b2 50°, consisting chiefly of I and PhMe, (b) 47 g. 6-acetoxy-4-methoxy-1-methylcyclohexene (III), b2 73-83°, and (c) 23 g., b2 83-120°, consisting of the starting material and more highly brominated products. Warming 92 g. III in 300 cc. 50% EtOH 3 hrs. at 60° with 31 g. KOH in 500 cc. EtOH and taking up the saponification product in 600 cc. CH2Cl2 gives 84.5% 6-hydroxy-4-methoxy-1-methylcyclohexene (IV), b4 82-4° (2-anthraquinonecarboxylate m. 170°). Adding slowly 160 g. tert-Bu chromate in 360 cc. anhydrous C6H6 to 50 g. IV, keeping the mixture 3 days at 20°, decomposing it in the cold with a saturated (CO2H)2 solution until **CO2** is no longer evolved, and extracting the aqueous layer with CH2Cl2 gives 69% 6-oxo-4-methoxy-1-methylcyclohexene (V), b10 90-2°, λmaximum 233 mμ, ε 7,800 (MeOH) (semicarbazone, m. 179.5-80°, λmaximum 263-4 mμ, ε 22,180; 2,4-dinitrophenylhydrazone, red needles, m. 172-3°), also obtained in 70-4% yield from 13 g. IV shaken with 130 g. MnO2 70 min. at 20° (phenylsemicarbazone, m. 163°). Passing dry CH:CH into 60 cc. ether and 500 cc. NH3 containing 4 g. Li until the solution is decolorized, then adding dropwise 17 g. V in 50 cc. ether, keeping the mixture 3 hrs., adding 200 cc. ether and the calculated amount of NH4Cl, and distilling the residue of the ether solution give 4 fractions:
(d) 6.5 g., b2 65°, chiefly o-MeC6H4OH (VI), (e) 3.6 g., b2 65-75° (VI and traces of 6-hydroxy-4-methoxy-1-methyl-6-ethinylcyclohexene (VII), (f) 2.3 g., b2 75-88°, consisting of VII and some VI, and (g) 4.4 g. residue. Redistn. of f gives 9.8% VII, b1.5 84-5.5°, clusters of needles, m. 54.5-5°. Adding dropwise 9.45 cc. MeLi in ether containing 52 mg. Li to 622 mg. VII in 7.5 cc. ether, refluxing the solution 40 min., then adding 1.03 g. C19-ketone (cf. W. and Grundmann, C.A. 30, 8225.6) in 7.5 cc. ether, refluxing the mixture 3 hrs., decomposing it with a saturated NH4Cl solution, and **chromatographically** fractionating the reaction product (1.65 g.) give 845 mg. of the compound C29H46O3 (VIII) as a very viscous oil which (2.578 g.) is treated in 15

cc. absolute MeOH with 2.3 g. Pd-C overnight, the solution filtered, the residue
washed with 25 cc. MeOH, the filtrate shaken with 400 mg. fresh 5% Pd-C (deactivated with a drop of quinoline) in a H atmospheric, and another 400 mg. poisoned Pd-C added, causing the absorption of 139 cc. H after 275 min. and giving the partially hydrogenated product (IX). Treating 220 mg. IX in 1.5 cc. ether 10 min. with 128 mg. P2I4 in 1.22 cc. CS2, shaking the ether solution with 2N NaOH and a saturated Na2S2O3 solution until colorless, and
working up the mixture in a N atmospheric give isotachysterol Me ether (X), which,
after **chromatographic** purification and distillation (b0.001 160-80°), shows λmaximum 280, 290, 302 mμ, ε 22,400, 27,200, 20,500; isotachysterol has λmaximum 290 mμ, ε 41,000. The reaction mechanism of these condensations is discussed.

IT Spectra
(of isotachysterol methyl ether and related compds.)

IT 2-Cyclohexen-1-ol, 1-{2-[hexahydro-4-hydroxy-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-indanyl]vinyl}-5-methoxy-2-methyl-
2-Cyclohexen-1-ol, 1-{[hexahydro-4-hydroxy-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-4-indanyl]ethynyl}-5-methoxy-2-methyl-
4-Indanol, hexahydro-4-[(1-hydroxy-5-methoxy-2-methyl-2-cyclohexen-1-yl)ethynyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-
4-Indanol, hexahydro-4-[2-(1-hydroxy-5-methoxy-2-methyl-2-cyclohexen-1-yl)vinyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-
Indan, 5,6,7,7a-tetrahydro-4-[2-(5-methoxy-2-methyl-1-cyclohexen-1-yl)vinyl]-7a-methyl-1-(1,4,5-trimethyl-2-hexenyl)-
Isotachysterol, methyl ether
Semicarbazide, 1-(5-methoxy-2-methyl-2-cyclohexen-1-ylidene)-4-phenyl-

IT 2-Cyclohexen-1-ol, 1-ethynyl-5-methoxy-2-methyl-
(and derivs.)

IT **50-14-6**, **Vitamin, D2**
(-related compds.)

IT 98558-30-6, 2-Cyclohexen-1-one, 5-methoxy-2-methyl-
(and derivs.)

IT 108420-17-3, 2-Cyclohexen-1-ol, 5-methoxy-2-methyl-
(and esters)

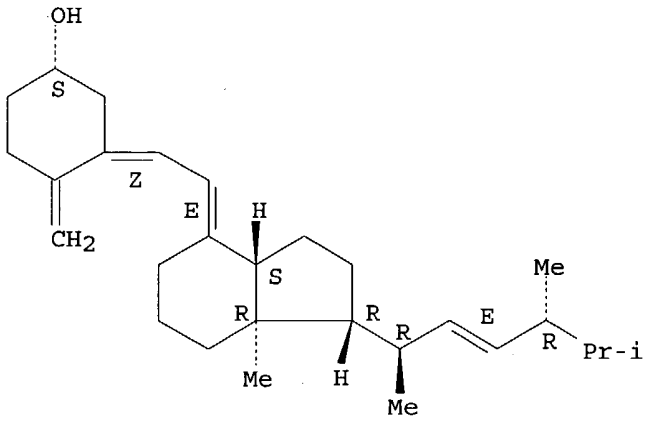
IT 98559-35-4, Ether, 5-bromo-4-methyl-3-cyclohexen-1-yl methyl
102878-81-9, Cyclohexanol, 4-methoxy-1-methyl- 108420-43-5, Ether, methyl 4-methyl-3-cyclohexen-1-yl 112116-88-8, 2-Anthraquinonecarboxylic acid, 5-methoxy-2-methyl-2-cyclohexen-1-yl ester
(preparation of)

IT **50-14-6**, **Vitamin, D2**
(-related compds.)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L79 ANSWER 28 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1953:44659 HCAPLUS
DN 47:44659
OREF 47:7531f-i,7532a-i
ED Entered STN: 22 Apr 2001
TI Synthesis of **provitamin D** with varied side chains
AU de Vries, Harmen; Backer, H. J.
CS Univ. Groningen, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1952**), 71, 719-32
CODEN: RTCPB4; ISSN: 0370-7539
DT Journal
LA French
CC 10 (Organic Chemistry)
OS CASREACT 47:44659
AB cf. C.A. 45, 3857g. 3β-Hydroxy-17-(1-methylpentyl)-Δ5,7-androstadiene (7-dehydrobisnorcholesterol) (I), the 17-(1-methylhexyl) homolog (7-dehydronorcholesterol) (II), 17-(1-methylheptyl) homolog (III), 17-(1-methyloctyl) homolog (IV), 17-(1-methyl-5-phenylpentyl) analog (V), and 3β-hydroxy-Δ5,7-androstadiene (VI), prepared from the corresponding Δ5-sterols, were **irradiated** and tested for antirachitic activity; only **irradiated** V showed a little activity. For the preparation of provitamins, all the reactions were carried out under N or **CO2**; for bromination and dehydrobromination all compds. and solvents were carefully dried; m.ps. were determined in evacuated capillaries, rotations in 2% CHCl3 solution free of HCl, absorption spectra in alc. purified with iodine and powdered Zn (3,5-dinitrobenzoates were taken up in CHCl3 and diluted with alc.). All Δ5,7-sterols showed absorption maximum at 262.1 (shoulder), 271.3, 281.8, and 293.4 mμ (molar extinctions (ε) are given for these 4 λs in the exptl. details); Δ4,6-isomers showed absorption maximum in the region 230-48 mμ. Bisnorcholesterol acetate (4.01 g.) in 50 cc. CCl4 refluxed with 2.14 g. N-bromosuccinimide (VII), **irradiated** ("biosol" lamp) several min. (orange-yellow solution), treated with 0.5 cc. s-collidine, filtered, the filtrate concentrated (20° in vacuo) with several cc. xylene added at the end, the residue taken up in 20 cc. xylene and 10 cc. collidine, heated 15 min. to 140°, concentrated, filtered, the filtrate heated 30 min. with MeOH-KOH, diluted with H2O, extracted with ether, and the extract washed with dilute H2SO4 and H2O gave 73% I as the 3,5-dinitrobenzoate, m. 160-6° (containing some Δ4,6-isomer), recrystd. from Me2CO-MeOH to give the pure compound, m. 193-4° (decomposition), [α]D -46.5°, ε at maximum 15501, 15503, 14275, and 8519, and saponified with MeOH-KOH in C6H6 to give pure I, m. 120-20.8°, [α]D -120°, ε at maximum 6576, 9548, 10069, and 5774;

acetate, m. 120.5-1.5°, [α] -79.3°, ε at maximum 7181, 10317, 10870, and 6201, resp. Similarly the following were prepared: II [from 10.37 g. norcholesteryl acetate with 5.34 g. VII, isolated in 62% yield as the 3,5-dinitrobenzoate, m. 194.5-5.5° (decomposition) (from Me2CO-CHCl3), [α]D -45.4°], m. 124-4.2° (from Me2CO-MeOH; heated in vacuo at 95°), [α]D -129.2°, ε at maximum 7605, 11020, 11640, and 6650; acetate, m. 111.5-12.5° (from Me2CO-alc.), ε at maximum 8033, 11424, 11940, and 6815. III [from 8.57 g. 3β-acetoxy-17-(1-methylheptyl)-Δ5-androstene with 4.27 g. VII, isolated in 35% yield as the 3,5-dinitrobenzoate, m. 198-9° (decomposition) (from CHCl3-Me2CO), [α]D -40.8°], m. 110.5-11° (meniscus formed at 120°) (from MeOH-Me2CO) (the diene from dilute Me2CO, dried at 85°, contained 0.5 mol. H2O), [α]D -124.5°, ε at maximum 7674, 10956, 11562, and 6564; acetate, m. 110.5-11° (from MeOH) (mixed with II gave marked lowering of m.p.), [α]D -75.6°, ε at maximum 8219, 11605, 12215, and 6986. IV, [from 8.854 g. 3β-acetoxy-17-(1-methyloctyl)-Δ5-androstene and 4.272 g. VII, purified via the 3,5-dinitrobenzoate, m. 209-9.5°, in 44% yield], m. 110-10.5° (from dilute Me2CO) (dried at 85° in vacuo, the crystals contained about 0.33 mol. H2O), [α]D -109.7°, ε at maximum 6916, 10032, 10566, and 6149; acetate, m. 94-6°, [α]D -67°, ε at maximum 6950, 9726, 10185, and 5846. V, [from 3β-acetoxy-17-(1-methyl-5-phenylpentyl)-Δ5-androstene and VII, isolated as the 3,5-dinitrobenzoate, m. 189-90° (decomposition), [α]D -34.7°], m. 76-8° (meniscus formed at 112°) (dried in vacuo at 55°, the crystals contained about 0.7 mol. H2O), [α]D -66°, ε at maximum 8058, 9831, 9925, and 5735. VI, [from 3β-acetoxy-Δ5-androstene (VIII) and VII, isolated in 42% yield as the 3,5-dinitrobenzoate, m. 195.5-6° (decomposition), [α]D -55°, ε at maximum 13527, 12738, 11446, and 7163], m. 157-8°, [α]D -206.6°, ε at maximum 6874, 9816, 10364, and 5886; acetate, m. 118.5-19°, ε at maximum 7418, 10144, 10594, and 6098. **Ultraviolet** absorption curves are given graphically. 3-Cholestanone dithioethylene ketal (96% from 3-cholestanone; cf. C.A. 44, 8931g), m. 144-4.6°, with Raney Ni gave cholestane, m. 80°. 3β-Acetoxy-Δ5-androsten-17-one, m. 170.5-1°, with (CH2SH)2 and HCl gave 91% dithioethylene ketal (IX), m. 188-9°, [α]D -88.8°; 4.07 g. IX refluxed 4 hrs. with 25 g. Raney Ni in MeOH gave 85% VIII, m. 95.5-7°. Attempts to make these provitamins by modification of the side chain of ergosterol (X) were unsuccessful because of the difficulties in protecting the conjugated double bond. X (5 g.) kept 24 hrs. at room temperature with 5 cc. Ac2O and 50 cc. absolute C5H5N gave X acetate (XI), m. 179.5° (from MeOH-C6H6). XI (4.3 g.) heated in a sealed tube 8 hrs. at 135° with 1.6 g. maleic anhydride (XII) in 10 cc. anhydrous xylene (cf. Inhoffen, C.A. 28, 1350.9), concentrated in vacuo, and the solid residue crystallized from ether gave 16% addition product (XIII), m. 217-18° (monoclinic crystals, β = 93.75°, a:b:c = 3.321:1:1.14), and some needles (sometimes allied with rosettes) (A), m. 174°. Concentration of the ether solution gave a mixture, m. 158-75°, which on crystallization from MeOH yielded needles (B), m. 206-7°, and platelets (C), m. 169-71°; A and B are probably isomers of XIII; C (with an absorption maximum at 251 mμ) is probably an isomer or a mixture of isomers of XI. Results of addns. of 2.28 millimoles XI and 2.84-9.74 millimoles XII, heated 8 hrs. in various solvents at 135-40°, are tabulated; in the best reaction 20% XIII was obtained with 6.84 millimoles XII in 4 cc. xylene. Dimethylmaleic anhydride, heated at 140° with XI gave no addition product. SO2 caused isomerization of XI without the formation of sulfones. XI (2.19 g.) in 10 cc. ether, saturated at 0° with SO2, heated 5 hrs. in a sealed tube at 100°, the solution washed, concentrated, and the semisolid residue crystallized from Me2CO, gave 0.9 g.

colorless needles, m. 126-7°, [α]D -101.8°, 0.7 g. solid, m. 125.2-6.2°, and 0.3 g. solid, m. 120°; these products, heated with Ac2O and separated **chromatographically** gave several fractions containing no S, probably isomers of XI, m. between 120-6°, which did not depress each other's m.p.; [α]D varied from -93 to -114°; absorption spectra maximum appeared at 251.5 mμ with a slight shoulder at 245 mμ. The Δ7,14-isomer of XI (0.5 g.) heated 7 hrs. with 1.5 g. XII in 10 cc. C6H6, concentrated, the residue saponified with MeOH-KOH, diluted with H2O, extracted with ether, the ether solution concentrated, and the residue (440 mg., with the same absorption spectrum as before reaction with XII), gave the acetate, m. 127.5°, [α]D -93°, λmaximum 251.3 mμ. The alkaline solution acidified and the product acetylated gave B, m. 205-6°.

IT Rickets
(antirachitic substances, from **provitamin D** derivs.)

IT Spectra
(of **provitamin D** derivs.)

IT 3-Cholestanone, cyclic ethylene mercaptole
5,7-Androstadien-3β-ol, 17-(1-methyl-5-phenylpentyl)-
5,7-Androstadien-3β-ol, 17-(1-methyl-5-phenylpentyl)-, 3,5-dinitrobenzoate
5,8-Ethano-15H-cyclopenta[a]phenanthrene-18,19-dicarboxylic anhydride, 1,2,3,4,9,10,11,12,13,14,16,17-dodecahydro-3-hydroxy-10,13-dimethyl-17-(1,4,5-trimethyl-2-hexenyl)-, acetate
5-Androsten-17-one, 3β-hydroxy-, cyclic ethylene mercaptole, acetate
5-Androsten-3β-ol, acetate
7,14,22-Ergostatrien-3-ol, acetate
7,14,22-Ergostatrien-3-ol, maleic anhydride adduct
Cholestane

IT 27-Nor-5,7-cholestadien-3β-ol
5,7-Androstadien-3β-ol
5,7-Androstadien-3β-ol, 17-(1-methylheptyl)-
5,7-Androstadien-3β-ol, 17-(1-methyloctyl)-
5,7-Androstadien-3β-ol, 17-(1-methylpentyl)-
Bisnorcholesterol, 7-dehydro-
Norcholesterol, 7-dehydro-
(and esters)

IT **Provitamin, D**
(derivs.)

IT Ergosterol, acetate, adduct with maleic anhydride
(isomers)

IT 108-31-6, Maleic anhydride
(adducts, with steroids)

IT 180-20-1, Spiro[3H-cyclopenta[a]phenanthrene-3,2'-[1,3]dithiolane]
187-02-0, Spiro[17H-cyclopenta[a]phenanthrene-17,2'-[1,3]dithiolane]
(derivs.)

L79 ANSWER 29 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1953:33261 HCAPLUS
DN 47:33261
OREF 47:5640a-d
ED Entered STN: 22 Apr 2001
TI Aqueous emulsions of lipide-soluble vitamins
IN Zentner, Margaret R.
PA **Hoffmann-La Roche Inc.**
DT Patent
LA Unavailable
CC 17 (Pharmaceuticals, Cosmetics, and Perfumes)
FAN.CNT 1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

PI US 2628930 19530217 US <--
AB A procedure is described for the preparation of aqueous emulsions of lipide-soluble
vitamin-active materials (A, D, E) which exhibit a high degree of chemical and phys. stability. Thus, warm 55 g. of α-tocopherol, 10 g. glyceryl monostearate (a fatty acid monoester having 12-18 C atoms in the acid radical), and 5 g. L-ascorbyl palmitate at 80-5° under a **CO2** atmospheric until a clear molten mass is obtained; add 750 cc. of a 4% gelatin solution (pH 8.5) which contains 30 g. gelatin, 1.35 g. Nipagin (Me p-hydroxybenzoate), 0.15 g. Nipasol (Pr p-hydroxybenzoate), and 0.375 g. NaOH. After the formation of a buff-colored emulsion add 50 cc. warm glycerol containing 0.45 g. Nipagin and 0.05 g. Nipasol. Then add 20 cc. water which contains 0.677 g. citric acid and 4.675 g. Na2HPO4. Next add 10 cc. water containing Na2SO3. Finally enough water is added to adjust the volume to 1 l. and enough NaOH to adjust the pH to about 7. Homogenize the emulsion for 20 min. at 3000 lb. pressure. The homogenized emulsion is ampuled and sterilized.
IT Vitamins
(aqueous emulsions of fat-soluble)
IT 1406-18-4, Vitamin E
(aqueous emulsions of lipide-soluble)
IT **1406-16-2, Vitamin D**
(emulsion containing)
IT 11103-57-4, Vitamin, A
(emulsions (aqueous) of)
IT **1406-16-2, Vitamin D**
(emulsion containing)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 30 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1953:32761 HCAPLUS
DN 47:32761
OREF 47:5567c-e
ED Entered STN: 22 Apr 2001
TI Experiences with the chemical determination of **vitamin D**. III
AU Diemair, W.; Manderscheid-Schwindling, G.
CS Univ. Frankfort a.M., Germany
SO Zeitschrift fuer Analytische Chemie (**1953**), 138, 1-8
CODEN: ZANCA8; ISSN: 0372-7920
DT Journal
LA Unavailable
CC 12 (Foods)
AB cf. C.A. 46, 10474e. Impurities in the SbCl3 reagent or traces of COCl2 in the CHCl3 may cause errors by changing the green color toward red. **Vitamin D** in **irradiated** milk must have the milk fat in an atmospheric of N or **CO2** because it is sensitive to the atmospheric The **chromatographic** determination is likely to be in error if the absorption tube is not filled uniformly with Al2O3. Sometimes enough eluant is not used. Care is necessary in separating the layers. From homogenized milk it is not easy to remove the butter fat. Exptl. data are given to illustrate these and other sources of error. These data should prove helpful to those engaged in testing pharmaceutical prepns., vitamin concentrates, ordinary milk, homogenized milk, and baby foods.
IT Milk
(analysis, determination of **vitamin D**)
IT Carcinogenic substances
(food dyes as)
IT **Chromatography and Adsorption analysis**
(of **vitamin D** in milks)

IT Vitamins
(prepns. and concentrates of, **vitamin D** determination in)
IT Pharmaceuticals
(**vitamin D** determination in)
IT **1406-16-2, Vitamin D**
(determination or assay of)
IT **1406-16-2, Vitamin D**
(determination or assay of)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L79 ANSWER 31 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1952:26697 HCAPLUS
DN 46:26697
OREF 46:4557f-i
ED Entered STN: 22 Apr 2001
TI A new homolog of **provitamin D3**
AU Strating, J.; Backer, H. J.
SO Proc. Koninkl. Nederland. Acad. Wetenschap. (**1951**), 54B, 13-15
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
AB The preparation of 7-dehydro-3-homocholesterol (I) is described. 7-Dehydrocholesteryl chloride (C.A. 45, 3858g) is transformed through its Grignard derivative and reaction with **CO2** into the 3-carboxylic acid (II) (m. 214-15° in vacuo, clear liquid at 260°). II gives with CH2N2 the Me ester (III), m. 118.5-19.5° (clear at 127°), [α]D20 -70.9° (CHCl3). Reduction of III with LiAlH4 gives I, m. 125-6° (in vacuo, slight alteration at 120°), [α]D20 -103.4° (CHCl3); acetate, m. 97.5-9°, [α]D18 -87.2°; benzoate, m. 98-100°, [α]D18 -59.9°; 3, 5-dinitrobenzoate, m. 183-4°, [α]D18 -24.3°. The **ultraviolet** spectra of I, II, and III are almost identical with that of 7-dehydrocholesteryl acetate, which proves that no rearrangement of the double bonds has taken place. I gives a precipitate with digitonin, showing that it possesses the β-configuration at C-3. Treatment of the Grignard compound of cholesteryl chloride with **CO2** and esterification of the acid formed with CH2N2 gives a Me ester (IV) with a sharp and constant m.p., 101.5-2.5°. Reduction of IV with LiAlH4 gives 3-homocholesterol (V), m. 129-30°, [α]D15 -35.5°, gives a digitonide, and is therefore the 3-β-form. The acetate of V can be transformed by bromination and dehydrobromination into a compound identical with the acetate of I, thus proving the stereochem. structure of the latter. The yields during the various steps of the synthesis of I are nearly quant.
IT Steroids
IT Spectra
(of steroids)
IT 3-Cholesterylcarboxylic acid
3-Cholesterylcarboxylic acid, methyl ester
3-Homocholesterol
3-Homocholesterol, acetate
5,7-Cholestadiene-3β-carboxylic acid
5,7-Cholestadiene-3β-carboxylic acid, methyl ester
5-Cholestene-3β-carboxylic acid
5-Cholestene-3β-methanol
5-Cholestene-3β-methanol, acetate
9(11)-Etioallocholenic acid, 3β-hydroxy-, methyl ester
9(11)-Etiocholenic acid, 3α-hydroxy-, methyl ester
9(11)-Etiocholenic acid, 3α-hydroxy-, methyl ester, acetate
IT 3-Homoprovitamin D3

(and derivs.)
IT 3-Homocholesterol, 7-dehydro-
5,7-Cholestadiene-3β-methanol
(and esters)
IT 5836-78-2, 5-Cholestene-3β-carboxylic acid, methyl ester
(preparation of)

L79 ANSWER 32 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1952:2773 HCAPLUS
DN 46:2773
OREF 46:517d-i,518a-d
ED Entered STN: 22 Apr 2001
TI Compounds related to **provitamin D3.** III.
Homocholesterol and the corresponding provitamin (3-homoprovitamin D3)
AU Strating, J.; Backer, H. J.
CS Univ. Groningen, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1951**
), 70, 389-402
CODEN: RTCPB4; ISSN: 0370-7539
DT Journal
LA English
CC 10 (Organic Chemistry)
AB cf. C.A. 45, 3859a. The term 3-homosterol is proposed for the new type of homology resulting when the 3-HO group of a sterol is replaced by a hydroxyalkyl group. The preparation and properties of 3-homocholesterol (3-β-hydroxymethyl-5-cholestene) (I) and the corresponding provitamin, 3-homoprovitamin D3 (3β-hydroxymethyl-5,7-cholestadiene) (II) are described. Me 5-cholestene-3β-carboxylate (III), prepared by esterification of 3-cholesterylcarboxylic acid with CH2N2 and separation of the epimers by recrystn. from MeOH, m. 101.5-2.5°. III with LiAlH4 in ether yields 99% I, colorless leaflets from EtOH, m. 129-30°, [α]D15 -35.5° (CHCl3); acetate (IV), colorless leaflets from EtOH, m. 83-4°, [α]D20 -25.7° (CHCl3); benzoate, leaflets from EtO-Ac, m. 158-9°, [α]D20 -24.7° (CHCl3); 3,5-dinitrobenzoate, yellowish needles from Me2CO, m. 195.5-6° (in vacuo), [α]D18 -19.6° (CHCl3); digitonide, rosettes of needles. Since pure I could be recovered from the mother liquor remaining from the digitonide precipitation and also by the decomposition of the recrystd. digitonide with anhydrous pyridine, it is concluded that I consists exclusively of the 3β-compound IV treated 5 min. with N-bromosuccinimide in boiling anhydrous CCl4 yields a complex mixture of products as a brown viscous mass from which no pure Br compound could be isolated. Elimination of HBr with anhydrous collidine at 140° in a **CO2** atmospheric (15 min.), followed by refluxing in 3% alc. KOH in a N atmospheric (30 min.) yields a brown half-solid residue, which on treatment with (O2N)2C6H3COCl in anhydrous pyridine, and addition of water yields a yellow precipitate of crude dinitrobenzoate, m. 184-5° after recrystn. from Me2CO, and representing a mixture of 50% of a 5,7- and 4% of a 4,6-cholestadiene (the remainder probably unchanged IV), as determined by the absorption spectrum. The yellow crystalline precipitate obtained from the collected filtrates by dilution with water was saponified with KOH-MeOH (N atmospheric) and then acetylated with Ac2O (**CO2** atmospheric); after recrystn. from MeOH, the material m. 63° forms an opaque melt at 71°, and a clear liquid at 80°. Absorption spectrum studies indicated a mixture of 46% of a 5,7- and 33.5% of a 4,6-cholestadiene. Further recrystn. from MeOH yields 3β-acetoxymethyl-5,7-cholestadiene (V), needles, m. 97-7.5° (in vacuo), [α]D18.5 -83.8° (CHCl3). Absorption spectrum analysis indicated a 2% contamination with a 4,6-cholestadiene. For the

preparation of II an alternate synthesis was chosen. Mg (2.06 g.) and 1.94 g. EtBr in 25 cc. anhydrous peroxide-free ether are heated and mixed prudently (5 hrs.) with 26.16 g. 7-dehydrocholesteryl chloride in 450 cc. ether, the mixture refluxed 20 hrs., concentrated to 175 cc., heated an addnl. 20 hrs. (all operations carried out in a N atmospheric), cooled to 0°, a stream of **CO2** led over the mixture 12 hrs., the ether solution decanted from the excess Mg, agitated with 10% H2SO4 in a **CO2** atmospheric, the precipitate collected, the residue obtained by evaporation of the filtrate digested with ether, the insol. portion added to the original precipitate, and the entire product recrystd. from C6H6, giving 9.64 g. 7-dehydro-3-cholesterylcarboxylic acid (VI), m. 214-15° (opaque melt), 260° (clear liquid) in vacuo. The anomalous m.p. is apparently not a consequence of impurity. VI with CH2N2 in ether yields 89% Me ester (VII), long needles from MeOH (**CO2** atmospheric), m. 118.5-19°, clear liquid 127° (in vacuo), [α]D20 -70.9° (CHCl3), easily soluble in light petroleum, ether, CHCl3, C6H6, less soluble in Me2CO, difficultly soluble in MeOH, EtOH. VII, on examination between crossed Nicols, did not show double refraction. VII with LiAlH4 in anhydrous ether yields a thick white precipitate which, after decomposition of the excess LiAlH4 with EtOAc, is dissolved with dilute H2SO4, the ether solution washed with water, dried, evaporated, and the residue recrystd. from MeOH, yielding II as fine white needles, m. 125-6° (slight change at 120°) in vacuo, [α]D20 -103.4° (CHCl3); digitonide, rosettes of needles; acetate, fine needles from MeOH, m. 97.5-9° (in vacuo), [α]D18 -87.2° (CHCl3); benzoate, fine needles from MeOH, m. 98-100° (in vacuo), [α]D18 -59.9° (CHCl3); 3,5-dinitrobenzoate, long yellow needles from Me2CO, m. 183-4° (decomposition) (in vacuo). Direct bromination of III, followed by elimination of HBr, gives a mixture from which no VII could be isolated. The absorption spectra of the compds. discussed, in absolute EtOH from 218-300 mμ, are given in tabular and graphical form. From the absorption curves, it appears that the mol. extinction for II is significantly higher (approx. 1%) than that for 7-dehydrocholesterol. A similar difference is noted in the spectra of the acetates. The phenomenon seems to be of a general character: the mol. extinction appears to increase with increase in mol. weight for a number of steroids with the conjugated system in positions 5-7.

IT Nomenclature
(` ` 3-homosterol'')
IT Spectra
(of steroids)
IT 5,7-Cholestadiene-3β-carboxylic acid
5,7-Cholestadiene-3β-carboxylic acid, methyl ester
IT 3-Homocholesterol
3-Homoprovitamin D3
5,7-Cholestadiene-3β-methanol
5-Cholestene-3β-methanol
(and derivs.)
IT 3-Homocholesterol, 7-dehydro-
(and esters)
IT Cholestenone-4-C14
(preparation of)
IT 3-Homosterol
(the term)
IT 11025-29-9, Digitonin, compound with cholesterol
(with steroids)

L79 ANSWER 33 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1951:21811 HCAPLUS
DN 45:21811
OREF 45:3859a-f
ED Entered STN: 22 Apr 2001

TI Compounds related to **provitamin D3**. II. The sulfur analog of **provitamin D3**
AU Strating, J.; Backer, H. J.
CS Univ., Groningen, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1950**), 69, 909-20
CODEN: RTCPB4; ISSN: 0370-7539
DT Journal
LA English
CC 10 (Organic Chemistry)
OS CASREACT 45:21811
AB The synthesis of 5,7-cholestadiene-3-thiol (I) is achieved by the reduction of 7-dehydrocholesteryl thiocyanate (II) or bis(7-dehydro-3-cholesteryl) disulfide (III) with LiAlH4. Cholesteryl thiocyanate (IV) is obtained in approx. 30% yield by refluxing 16 g. dry cholesteryl p-toluenesulfonate (V) and 5.6 g. KCNS in 100 cc. anhydrous MeCOEt 12 hrs., filtering the cooled reaction mixture, evaporating the filtrate under reduced pressure, and extracting the residual product several times with small quantities of Me2CO and crystallizing it from alc. to give pure IV, m. 127-8°. Me2CO proved to be an unsuitable medium for the above reaction (cf. Muller and B.acte.atyka, C.A. 35, 6243.7), as the presence of traces of water apparently caused the hydrolysis of V with production of di-cholesteryl ether as the only isolable material. Direct reduction of IV with LiAlH4 in ether affords 81% 5-cholestene-3-thiol (VI), colorless needles from dilute alc., m. 98-9.5°. Preparation of I by the attempted bromination of S-acetyl-5-cholestene-3-thiol (VII), m. 124-5°, [α]20.5D -52.1° (CHCl3), and the subsequent elimination of HBr proved unsuccessful. A portion of VII reacts with more than 1 Br atom/mol. so that a major portion of VII remains unchanged. Further difficulty was anticipated in the hydrolysis of S-acetyl-7-dehydro-5-cholestene-3-thiol, as VII could not be successfully saponified to the pure thiol VI. Saponification of VII with KOH in MeOH and C6H6 at room temperature yields dicholesteryl disulfide (VIII) and VI; saponification with a hot (65°) solution of KOH in an evacuated tube yields cholesterol and VI; and saponification with KSH in hot MeOH in an evacuated tube yields VIII and dicholesteryl trisulfide. Crude 7-bromocholesteryl thiocyanate, m. 108-11°, prepared from IV with N-bromosuccinimide, is readily converted to II by heating 20 min. at 140° with anhydrous collidine in a **CO2** atmospheric The product, after recrystn. from alc., m. 139-140.5° (in vacuo), [α]19.5D -39.6° (CHCl3). III is obtained by heating II with shaking for 9 hrs. at 90° with NaOEt in an air-free sealed tube; 4 cc. of 2 N HCl and 25 cc. each of C6H6 and ether are then added to the mixture, the nonaq. extract washed acid-free with water, dried over Na2SO4, filtered, the solvent removed under reduced pressure, and the residue washed with hot EtOH and recrystd. from AcOEt to yield III, rosettes of needles, m. 161-3°, [α]19D -12.8° (CHCl3). II with ethereal LiAlH4 furnishes 67% I; similar reduction of III gives 30% I. I, long needles from EtOH, m. 132-5°, [α]17.5D -62.2° (CHCl3). The **ultraviolet** absorption values for the 2 samples of I prepared from II and III are practically identical and compare closely with those for 7-dehydrocholesterol.
IT Spectra
(of 5,7-cholestadien-3β-ol related compds.)
IT 5,7-Cholestadiene-3-thiol
5-Cholestene-3-thiol
5-Cholestene-3-thiol, acetate
Cholesterol, thiocyanate
Cholesteryl thiocyanate, 7-bromo-
Cholesteryl trisulfide
Disulfide, bis(7-dehydrocholesteryl)

Thiocyanic acid, 5,7-cholestadien-3-yl ester
Thiocyanic acid, 7-bromocholesteryl ester
Thiocyanic acid, cholesteryl esters
IT 2469-23-0, Cholesteryl ether 39879-87-3, Cholesteryl disulfide
(preparation of)

L79 ANSWER 34 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1951:21810 HCAPLUS
DN 45:21810
OREF 45:3858g-i,3859a
ED Entered STN: 22 Apr 2001
TI Compounds related to **provitamin D3.** I.
7-Dehydrocholesteryl chloride and bromide
AU Strating, J.; Backer, H. J.
CS Univ., Groningen, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1950**), 69, 904-8
CODEN: RTCPB4; ISSN: 0370-7539
DT Journal
LA English
CC 10 (Organic Chemistry)
OS CASREACT 45:21810
AB cf. Bide, et al., C.A. 43, 3019i; Bernstein, et al., C.A. 43, 4679a.
7-Dehydrocholesteryl chloride (I) is prepared by the elimination of HBr from 7-bromocholesteryl chloride (II). II is obtained in 37% yield by heating an **irradiated** (Hg-arc lamp) mixture of cholesteryl chloride and N-bromosuccinimide in anhydrous CCl4 on a water bath for 15 min. The succinimide is filtered off and the filtrate is evaporated in vacuo at room temperature; II, after repeated recrystn. from anhydrous Me2CO, m. 133.5-4.5° (decomposition). HBr is eliminated by heating II in a **CO2** atmospheric with anhydrous collidine; the temperature of the bath is raised to 140° over a period of 30 min. and then maintained at that point an addnl. 20 min. The cooled mixture is extracted with peroxide-free ether and with dilute H2SO4, the washed and dried ether extract evaporated in vacuo at room temperature, and the viscous residue recrystd. from Me2CO, giving 20% I as fine needles, m. 133.5-4° (in vacuo). 7-Bromocholesteryl chloride (III), m. 135.8-6.2°, and 7-dehydrocholesteryl bromide (IV), m. 147-7.5° (in vacuo), are similarly obtained from cholesteryl bromide in slightly lower yields. The agreement of the absorption spectra of I and IV with that of 7-dehydrocholesterol demonstrates that the synthetic compds. have the same system of conjugated double bonds as is present in **provitamin D.**
IT Spectra
(of 5,7-cholestadien-3β-ol related compds.)
IT 5-Cholestene, 3β,7-dibromo-
5-Cholestene, 7-bromo-3β-chloro-

L79 ANSWER 35 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1937:53855 HCAPLUS
DN 31:53855
OREF 31:7490b-d
ED Entered STN: 16 Dec 2001
TI Chemical activation of sterols. III. Chemical activation of cholesterol
AU Eck, John C.; Thomas, Byron H.
SO Journal of Biological Chemistry (**1937**), 119, 621-30
CODEN: JBCHA3; ISSN: 0021-9258
DT Journal
LA Unavailable
CC 11E (Biological Chemistry: Nutrition)
AB cf. C. A. 31, 2264.4. Cholesterol when heated with H2SO4, sulfoacetic acid, fuming H2SO4, or chlorosulfonic acid in AcOH solution acquires

antirachitic properties. Maximum antirachitic potency was produced by using the proportions of 0.0025 mol. Ac2O, 0.002 mol. concentrated H2SO4 and 0.001 mol. cholesterol in glacial AcOH and heating at 85-90° for 3 hrs. SO2 and **CO2** were evolved in side reactions, the amount of SO2 produced not being correlated with the activity of the product. **Radiation** of the product did not affect the potency. Hence the product is not a **provitamin D** activatable by **ultraviolet radiation. Provitamin D** of heated purified cholesterol is not the precursor of the chemically activated product.

IT Sterols
(activation of)

IT Rickets
(antirachitic action, of cholesterol chemically activated)

IT 57-88-5, Cholesterol
(activation of)

L79 ANSWER 36 OF 36 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1937:10440 HCAPLUS
DN 31:10440
OREF 31:1421f-i,1422a-g
ED Entered STN: 16 Dec 2001
TI Concentration of **vitamin D** from tunafish liver oil
AU Neracher, O.; Reichstein, T.
SO Helvetica Chimica Acta (**1936**), 19, 1382-91
CODEN: HCACAV; ISSN: 0018-019X
DT Journal
LA Unavailable
CC 10 (Organic Chemistry)
AB The natural **vitamin D** from fish oils is not identical with calciferol (**vitamin D2**) in that the same amount of the former, expressed in rat units, has a greater effect on chicks than the latter (cf. Brockmann, C. A. 30, 6423.1). Accordingly, work on this problem has been discontinued but details for a simple and reproducible method of preparing concentrates (20%) containing about 5000 international units per mg. are given. Treatment with 3,5-(O2N)2C6H3COCl gave crystalline derivs. whose parent alcs. proved to be biologically inactive. The saponification of 1 kg. of tunafish oil with 300 g. KOH in 150 cc. air-free H2O and 1.3 l. of MeOH (distilled over KOH under N2) for 1.5 hrs. under strictly O2-free conditions, cooling, dilution with 4 l. of N2-saturated H2O and extraction with Et2O in a N2 atmospheric, gave 144.5 g. of yellow, partly crystalline unsaponifiable material (I). When not in process this material, like all other intermediates, was preserved in ampoules under vacuum. The solution of I in 400 cc. MeOH was cooled in a **CO2** atmospheric to 0°. The main portion of cholesterol crystallized out and gave 48.7 g. of crude oil with a biol. activity of less than 20 units per mg. The MeOH solution was then cooled with solid **CO2** in a Dewar vessel to -80° and after 30 min. the solid crystalline mass was filtered off through a 3-cm. thick, finely-powdered, compressed layer of solid **CO2** and washed with MeOH at -80°. The thawed residue was filtered and after vacuum drying gave 60.0 mg. of a cholesterol fraction (II) as a brown, partially crystalline oil with a D activity of about 1000 units and a vitamin A content of about 520 units per mg. The MeOH washings yielded 35.2 g. of a dark red-brown oil with 250 and about 1333 international units of D and A per mg., resp. Gradual cooling to -15° of a pentane solution of 30 g. II produced 8 g. of white crystals, m. 146°, which showed no A or D color reactions. A solution of 22 g. II in 200 cc. pentane was run, under **CO2** pressure, through a 60-cm. long, 45-mm. wide **chromatographic** column containing 500 g. of washed Al2O3 (Merck, according to Brockmann). The **chromatogram** was washed with 2 l. of pentane and yielded 2.608 g. of fluorescent oil with less than 10 units

D per mg. Washing with absolute C6H6 and Et2O (freshly distilled over CaCl2) gave 5.872 g. of material containing 2000 units D per mg. Elution with a 1:1 mixture of Et2O and MeOH yielded 11.682 g. of a strongly cholesterol-containing material with about 250 units D per mg. The column retained 1.838 g. of highly colored impurities. The above C6H6-Et2O and Et2O-MeOH eluates were separated by C6H4(CO)2O according to Ender's method (C. A. 27,523) into 12.7 g. of pentane-soluble alc., 0.569 g. of pentane-insol. alc. and 3.67 of inactive alc.-free material. The 12.7 g. was treated with 30 g. digitonin in 2 l. of 90% alc. and produced 8.48 g. of cholesterol-free alc. which was **chromatographed.** Elution gave a C6H6 filtrate containing 3.05 g. of material with a D activity of 6000 units per mg. and an Et2O filtrate containing 3.13 g. with 5000 units per mg. This latter material (3.025 g.) was dissolved in 10 cc. anhydrous pyridine and refluxed with 4 g. of 3,5-(O2N)2C6H3COCl in 6 cc. absolute C6H6 for 5 min. The cooled reaction mixture was precipitated with Et2O, filtered and washed with Et2O. After acidification with ice-cold HCl, neutralization, washing, and drying, the Et2O was distilled off and the residue was taken up in pentane. The solution was **chromatographed** and eluted with a pentane-C6H6 mixture with increasing C6H6 content to pure C6H6, then with C6H6-Et2O, pure Et2O and finally Et2O-Me2CO mixts. Fractions 4-6 of the 12 elutates yielded 500 mg. of flesh-colored needles of dinitrobenzoate I (III), C32H40N2O7, m. 202° (corrected). A solution of 50 mg. III in 16 cc. C6H6 was reductively saponified by heating for 15 min. with a solution of 200 mg. SnCl2 (treated with 50% KOH to slight turbidity) in 10 cc. absolute alc. The diluted reaction mixture was extracted with Et2O and yielded 34 mg. of a biologically inactive oily alc. which could be reconverted to the original III. The concentrated mother liquors from III gave, on long standing in pentane, 280 mg. of yellow needles of dinitrobenzoate II, C27H33N2O6, m. 181.5-2.5° (corrected), corresponding to an alc., C20H30-2O, which proved to be biologically inactive. The residual mother liquors were purified through the α-C10H7NH2 compound (cf. C. A. 21, 37) and yielded a small amount of a dinitrobenzoate III, m. 113°, from which an inactive alc. was obtained. The above alkaline stannite saponification avoids the oxidation action of the NO2 groups in the usual alkaline saponification of the dinitrobenzoates. Attempts to purify the concentrates by distillation at 0.0001 mm. at 150° gave 75% distillates. The cholesterol in tunafish liver oil is accompanied by a relatively rich amount of material precipitated with digitonin and showing a strong absorption band at about 250 mμ which may be provitamin D.

IT Oils
(tunny-liver, **vitamin D** from)
IT **1406-16-2, Vitamin D**
(preparation from tuna-fish-liver oil)
IT **1406-16-2, Vitamin D**
(preparation from tuna-fish-liver oil)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

=> => d all hitstr tot

L96 ANSWER 1 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **2001:343243** HCAPLUS
DN **135:131453**
ED Entered STN: 14 May 2001
TI Inherent possibilities of improving recovery and selectivity using a long solid phase trap in analytical **supercritical** fluid extraction

AU Eskilsson, Cecilia Sparr; Turner, Charlotta; Esbjornsson, Annelle; Mathiasson, Lennart
CS Department of Analytical Chemistry, Lund University, Lund, S-221 00, Swed.
SO Journal of Separation Science (2001), 24(4), 297-303
CODEN: JSSCCJ
PB Wiley-VCH Verlag GmbH
DT Journal
LA English
CC 80-4 (Organic Analytical Chemistry)
Section cross-reference(s): 17, 45
AB Possibilities of improving recovery and selectivity in **supercrit**. fluid extraction systems based on solid phase collection by using a longer trap (5 + 200 mm) instead of a standard trap (5 + 55 mm) were studied. Such a long trap may give higher sample capacity, and filled with a **chromatog.** trapping material it gives a column with higher separation efficiency than the standard trap. The system was tested on thermally labile substances such as fat-soluble vitamins and aromatic amines. Quant. recoveries of both vitamins and amines were obtained when using the long trap combined with modifier in the extracting fluid and trapping temps. below the b.p. of the modifier conditions that usually cause breakthrough losses of analytes. Also, the vitamins could be eluted from the long trap well separated from the lipid fraction, while this was not possible using the standard trap. The possibility of obtaining selectivity between substances of similar structures, such as aromatic amines, was studied on different types of trapping material (ODS, NH2, and CN). Fairly good fractionation was achieved using CN. This opens up the possibility to decrease the total anal. time by eluting the samples in fractions.
ST solid phase long trap **supercrit** fluid extn; vitamin fat soluble **supercrit** extn solid phase long trap; arom amine **supercrit** fluid extn solid phase long trap
IT Amines, analysis
RL: ANT (Analyte); PEP (Physical, engineering or chemical process); PRP (Properties); ANST (Analytical study); PROC (Process)
(aromatic, analytes; inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)
IT Leather
(dyed, samples; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for aromatic amines in)
IT Vitamins
RL: ANT (Analyte); PEP (Physical, engineering or chemical process); PRP (Properties); ANST (Analytical study); PROC (Process)
(fat-soluble, analytes; inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)
IT Fats and Glyceridic oils, analysis
RL: AMX (Analytical matrix); ANST (Analytical study)
(fish-liver, samples; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. of)
IT Trapping apparatus
(inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)
IT **Silica gel**, analysis
RL: ARU (Analytical role, unclassified); NUU (Other use, unclassified); ANST (Analytical study); USES (Uses)
(reaction products, octadecyl-, aminopropyl-, or cyanopropyl-modified, trapping materials; inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)
IT Rape oil
RL: AMX (Analytical matrix); ANST (Analytical study)

(samples; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. of)
IT Extraction
(**supercrit.**; inherent possibilities of improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction)
IT 59-02-9, α-Tocopherol 68-26-8, vitamin A 79-81-2, Retinyl palmitate 91-94-1, 3,3'-Dichlorobenzidine 95-53-4, o-Toluidine, analysis 119-13-1, δ-Tocopherol 119-90-4, 3,3'-Dimethoxybenzidine 148-03-8, β-Tocopherol **1406-16-2**, **vitamin D** 1406-18-4, vitamin E 7616-22-0, γ-Tocopherol
RL: ANT (Analyte); ANST (Analytical study)
(analyte; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)
IT 67-56-1, Methanol, analysis 67-63-0, 2-Propanol, analysis
RL: ARU (Analytical role, unclassified); NUU (Other use, unclassified); ANST (Analytical study); USES (Uses)
(mobile phase containing; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)
RE.CNT 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD
RE
(1) Berg, H; Biophys Methods 2000, V43, P391 HCAPLUS
(2) Bjorklund, E; J Liq Chrom & Rel Technol 2001
(3) Bowadt, S; J Chromatogr A 1994, V662, P424 HCAPLUS
(4) Jaremo, M; J Chromatogr A 2000, V877, P167 HCAPLUS
(5) Koinecke, A; J Chromatogr A 1997, V786, P155 HCAPLUS
(6) Lehotay, S; J Chromatogr A 1997, V765, P69 HCAPLUS
(7) Magard, M; J Agric Food Chem 1995, V43, P114
(8) Mannila, M; Organohalogen Compd 1998, V35, P137 HCAPLUS
(9) Mathiasson, L; submitted to Food Addit Contam 2001
(10) Mulcahey, L; Anal Chem 1992, V64, P2352 HCAPLUS
(11) Taylor, S; J Chromatogr Sci 2000, V38, P91 HCAPLUS
(12) Turner, C; J Agric Food Chem 2001, V49, P553 HCAPLUS
(13) van Bavel, B; Anal Chem 1996, V68, P1279 HCAPLUS
IT **1406-16-2, vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(analyte; improving recovery and selectivity using a long solid phase trap in anal. **supercrit.** fluid extraction for anal. for)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

L96 ANSWER 2 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1998:352383** HCAPLUS
DN **128:319006**
ED Entered STN: 10 Jun 1998
TI Statistical analysis of liquid trapping efficiencies of fat-soluble vitamins following **supercritical** fluid extraction
AU McDaniel, Lori H.; Long, Gary L.; Taylor, Larry T.
CS Virginia Polytechnic Institute, Department Chemistry, Virginia State University, Blacksburg, VA, 24061, USA
SO Journal of High Resolution Chromatography (1998), 21(4), 245-251
CODEN: JHRCE7; ISSN: 0935-6304
PB Huethig GmbH
DT Journal
LA English
CC 9-9 (Biochemical Methods)
Section cross-reference(s): 68
AB A series of 4 complete factorial expts. were performed to determine the major parameters affecting the trapping efficiencies of fat-soluble vitamins after

supercrit. fluid extraction The parameters varied were the collection solvent, extraction flow rate, collection temperature, restrictor temperature, and collection pressurization. The identity of the collection solvent had the most profound effect. The viscosity and surface tension of the collection solvent had great significance.

ST **supercrit** fluid extn vitamin trapping efficiency; vitamin fat soluble **supercrit** fluid extn

IT Vitamins
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(fat-soluble; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction studied by statistical anal.)

IT Surface tension
(liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction in relation to surface tension of collection solvent)

IT Viscosity
(liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction in relation to viscosity of collection solvent)

IT Solvents
(organic, collection; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction in relation to physico-chemical properties of)

IT Extraction
(**supercrit.**; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction studied by statistical anal.)

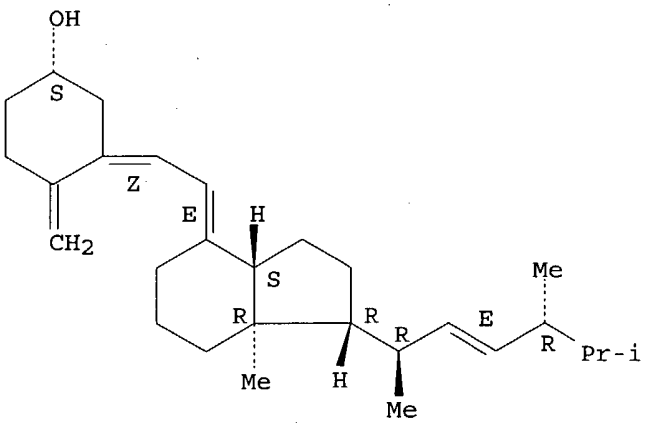
IT 64-17-5, Ethanol, uses 67-63-0, Isopropanol, uses 110-54-3, Hexane, uses 111-70-6, 1-Heptanol
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
(collection solvent; liquid trapping efficiencies of fat-soluble vitamins in **supercrit.** fluid extraction in relation to physico-chemical properties of)

IT **50-14-6, Vitamin d2** 59-02-9, α-Tocopherol **67-97-0, Cholecalciferol** 68-26-8, all-trans-Retinol 12001-79-5, Vitamin K
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction studied by statistical anal.)

IT **50-14-6, Vitamin d2 67-97-0, Cholecalciferol**
RL: PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)
(liquid trapping efficiencies of fat-soluble vitamins in **supercrit** . fluid extraction studied by statistical anal.)

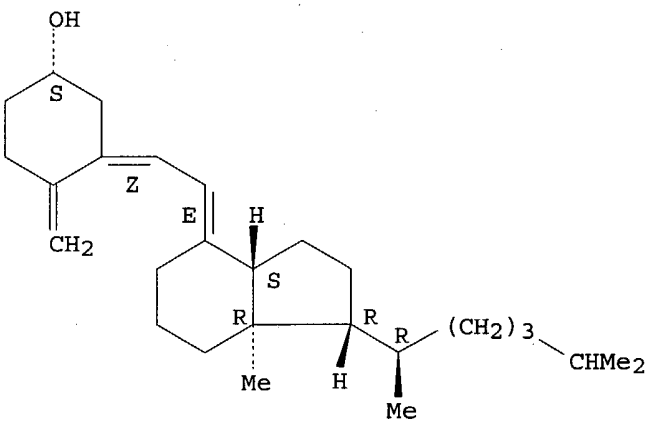
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 3 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1995:862751** HCAPLUS
DN **123:358081**
ED Entered STN: 18 Oct 1995
TI Online SFE-SFC coupling using micropacked columns
AU Ibanez, Elena; Herraiz, Marta; Reglero, Guillermo
CS Instituto de Fermentaciones Industriales, CSIC, Madrid, 28006, Spain
SO Journal of High Resolution Chromatography (1995), 18(8), 507-9
CODEN: JHRCE7; ISSN: 0935-6304
PB Huethig
DT Journal
LA English
CC 80-2 (Organic Analytical Chemistry)
AB A dynamic online coupled system of **supercrit.** fluid extraction (SFE) and **supercrit.** fluid **chromatog.** (SFC) based on micropacked columns was developed, and the performance of the SFE-SFC system was studied by extraction and anal. of a standard solution of lipid-soluble **vitamins** (**vitamin** A, A acetate, D2, **D3**, E, E acetate, K1, and K3) . The use of the micropacked columns enabled SFE-SFC

coupling by an device operating with only 1 pump and a 6-way switching valve integrated into the SF **chromatog.** Data on the performance of the SFE-SFC system are given, e.g. the retention times were reproducible and low detection limits were obtained.

ST **supercrit** fluid **chromatog** extn micropacked column

IT **Chromatography, supercritical** fluid
(**supercrit.** fluid **chromatog.-supercrit.** fluid extraction coupling with micropacked columns)

IT Extraction
(**supercrit.**, **supercrit.** fluid **chromatog.-supercrit.** fluid extraction coupling with micropacked columns)

L96 ANSWER 4 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN

AN **1995:823830** HCAPLUS

DN **123:226193**

ED Entered STN: 30 Sep 1995

TI Optimization of Separation of Fat-Soluble Vitamins by **Supercritical** Fluid **Chromatography** Using Serial Micropacked Columns

AU Ibanez, Elena; Tabera, Javier; Reglero, Guillermo; Herraiz, Marta

CS Instituto de Fermentaciones Industriales, CSIC, Madrid, 28006, Spain

SO Journal of Agricultural and Food Chemistry (1995), 43(10), 2667-71
CODEN: JAFCAU; ISSN: 0021-8561

PB American Chemical Society

DT Journal

LA English

CC 17-1 (Food and Feed Chemistry)

AB The advantages of using two online coupled micropacked columns in **supercrit.** fluid **chromatog.** for analyzing fat-soluble vitamins are evaluated. A rotatable central composite exptl. design is used to optimize the combination of some exptl. variables (temperature gradient and pressure gradient) involved in the **chromatog.** separation Relative standard deviations obtained under the conditions of the exptl. design giving the highest response are also included. The anal. of fat-soluble vitamins occurring in real-life samples (a pharmaceutical preparation for newborns and an infant formula) is presented.

ST vitamin **supercrit** fluid **chromatog** infant formula

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, optimization of separation of fat-soluble vitamins by **supercrit.** fluid **chromatog.** using serial micropacked columns)

IT Milk substitutes
(human, optimization of separation of fat-soluble vitamins by **supercrit**. fluid **chromatog.** using serial micropacked columns)

IT **112267-91-1**, Protovit
RL: AMX (Analytical matrix); ANST (Analytical study)
(optimization of separation of fat-soluble vitamins by **supercrit.** fluid **chromatog.** using serial micropacked columns)

IT **50-14-6**, **Vitamin D2** 58-27-5, Vitamin K3
58-95-7, Vitamin E acetate **67-97-0**, **Vitamin D3**
68-26-8, Vitamin A 1406-18-4, Vitamin E 11104-38-4, Vitamin K1
RL: ANT (Analyte); ANST (Analytical study)
(optimization of separation of fat-soluble **vitamins** by **supercrit.** fluid **chromatog.** using serial micropacked columns)

IT **112267-91-1**, Protovit
RL: AMX (Analytical matrix); ANST (Analytical study)
(optimization of separation of fat-soluble vitamins by **supercrit.** fluid **chromatog.** using serial micropacked columns)

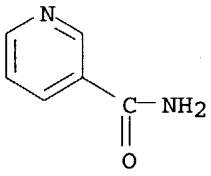
RN 112267-91-1 HCAPLUS

CN L-Ascorbic acid, mixt. with 3-[(4-amino-2-methyl-5-pyrimidinyl)methyl]-5-

(2-hydroxyethyl)-4-methylthiazolium chloride, (R)-2,4-dihydroxy-N-(3-hydroxypropyl)-3,3-dimethylbutanamide, [3aS-(3aα,4β,6aα)]-hexahydro-2-oxo-1H-thieno[3,4-d]imidazole-4-pentanoic acid, 5-hydroxy-6-methyl-3,4-pyridinedimethanol, 3-pyridinecarboxamide, retinol, riboflavin and (3β,5Z,7E,22E)-9,10-secoergosta-5,7,10(19),22-tetraen-3-ol (9CI) (CA INDEX NAME)

CM 1

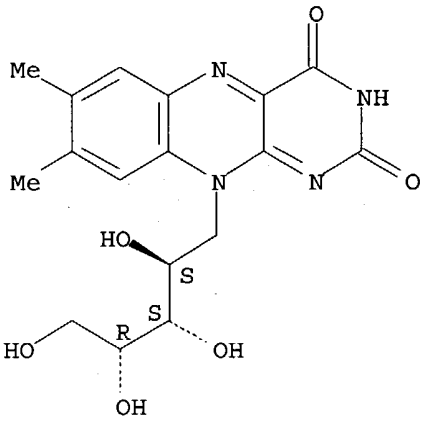
CRN 98-92-0
CMF C6 H6 N2 O



CM 2

CRN 83-88-5
CMF C17 H20 N4 O6

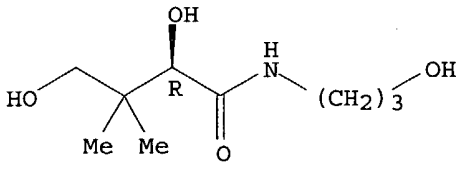
Absolute stereochemistry.



CM 3

CRN 81-13-0
CMF C9 H19 N O4

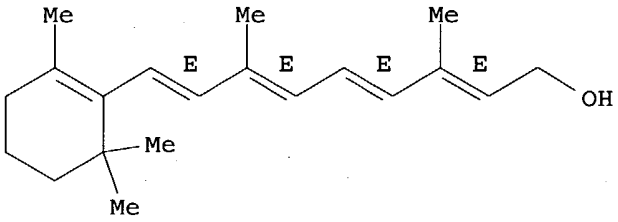
Absolute stereochemistry.



CM 4

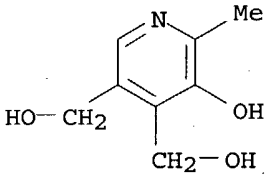
CRN 68-26-8
CMF C20 H30 O

Double bond geometry as shown.



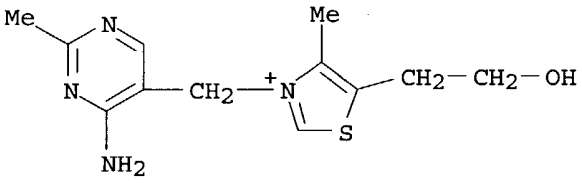
CM 5

CRN 65-23-6
CMF C8 H11 N O3



CM 6

CRN 59-43-8
CMF C12 H17 N4 O S . Cl

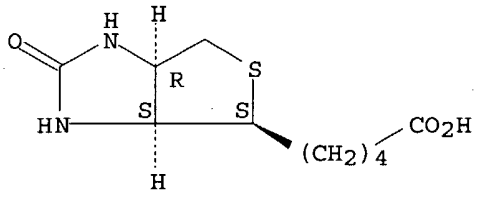


● Cl⁻

CM 7

CRN 58-85-5
CMF C10 H16 N2 O3 S

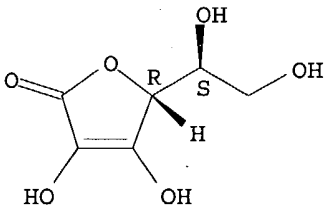
Absolute stereochemistry. Rotation (+).



CM 8

CRN 50-81-7
CMF C6 H8 O6

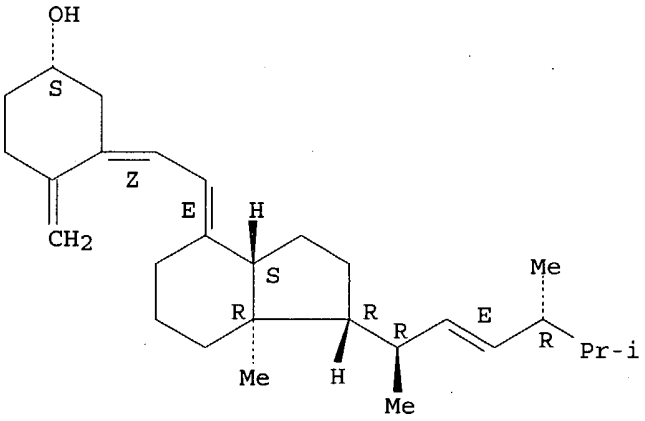
Absolute stereochemistry.



CM 9

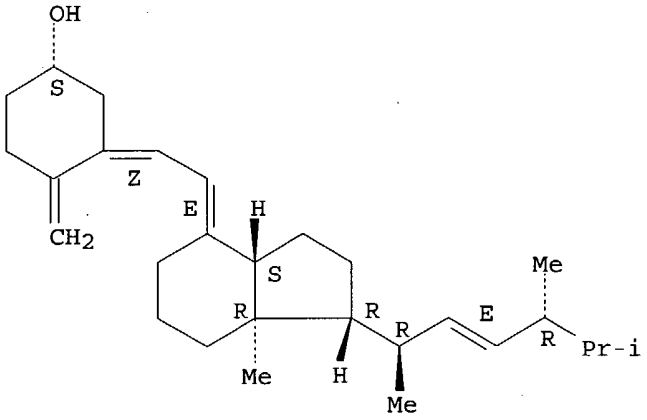
CRN 50-14-6
CMF C28 H44 O

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



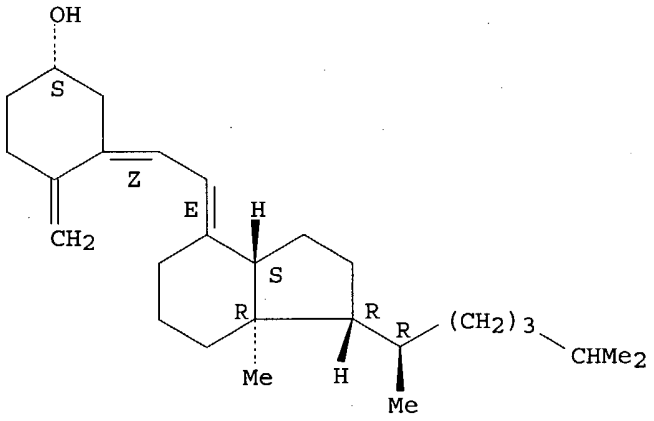
IT **50-14-6, Vitamin D2 67-97-0, Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(optimization of separation of fat-soluble **vitamins** by **supercrit.** fluid **chromatog.** using serial micropacked columns)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 5 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1995:549051** HCAPLUS
DN **122:274206**
ED Entered STN: 13 May 1995
TI Optimization of fat-soluble vitamin separation by **supercritical** fluid **chromatography**
AU Ibanez, E.; Tabera, J.; Reglero, G.; Herraiz, M.
CS Inst. Ferment. Ind., CSIC, Madrid, 28006, Spain
SO Chromatographia (1995), 40(7/8), 448-52
CODEN: CHRGB7; ISSN: 0009-5893
PB Vieweg
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)
AB Fat-soluble vitamin separation, achievable using a micropacked SFC column loaded with CW 20M, is optimized using a rotatable central composite exptl.

design. Two **chromatog.** response functions, based on relative retention and the number of peaks resolved are proposed. Temperature and pressure gradients giving the best response in the exptl. region are obtained and the effect of their variation on separation is evaluated. Relative standard deviations resulting from both absolute and normalized peak areas are also given.

ST fat soluble vitamin sepn **supercrit chromatog**

IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble; optimization of fat-soluble vitamin separation by **supercrit**. fluid **chromatog.**)

IT **Chromatography, supercritical** fluid
Pharmaceutical analysis
(optimization of fat-soluble vitamin separation by **supercrit.** fluid **chromatog.**)

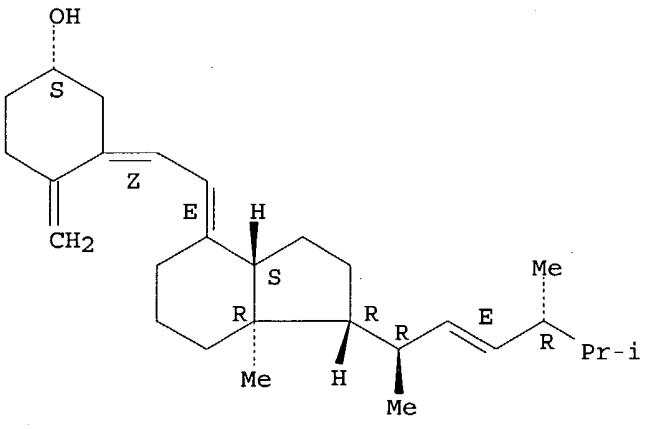
IT **50-14-6, Vitamin D2** 58-27-5, Vitamin K3 58-95-7, Vitamin E acetate 59-02-9 **67-97-0, Vitamin D3** 68-26-8, Vitamin A 127-47-9, Retinol acetate 11104-38-4, Vitamin K1
RL: ANT (Analyte); ANST (Analytical study)
(optimization of fat-soluble **vitamin** separation by **supercrit**. fluid **chromatog.**)

IT **50-14-6, Vitamin D2 67-97-0, Vitamin D3**
RL: ANT (Analyte); ANST (Analytical study)
(optimization of fat-soluble **vitamin** separation by **supercrit**. fluid **chromatog.**)

RN 50-14-6 HCAPLUS

CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

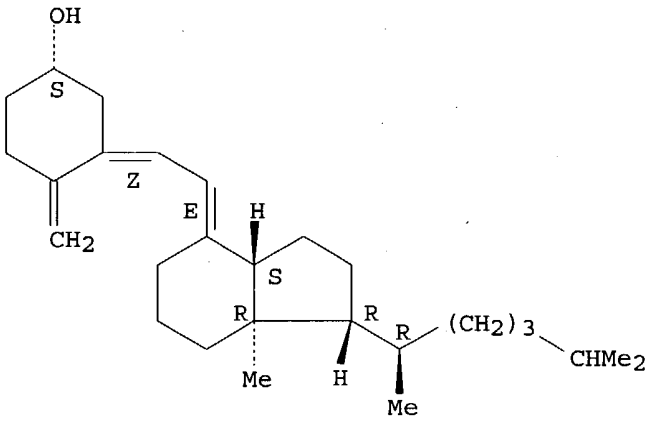
Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS

CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

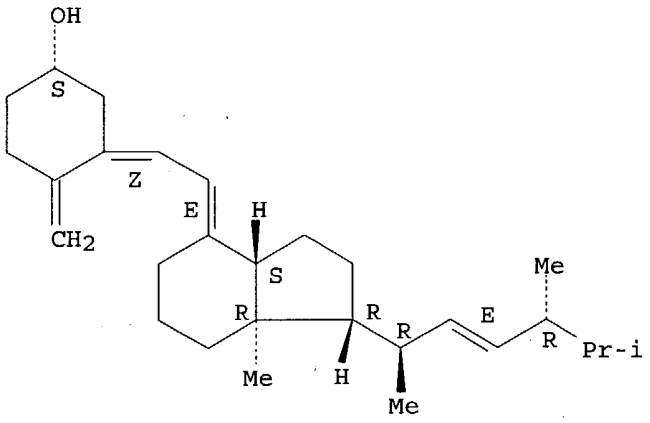
Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 6 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1988:479795** HCAPLUS
DN **109:79795**
ED Entered STN: 02 Sep 1988
TI Analysis of pharmaceuticals and other solutes of biochemical importance by **supercritical** fluid **chromatography**
AU White, C. M.; Gere, D. R.; Boyer, D.; Pacholec, F.; Wong, L. K.
CS SFC Res. Cent., Suprex Corp., Pittsburgh, PA, 15238, USA
SO HRC & CC, Journal of High Resolution Chromatography and Chromatography Communications (1988), 11(1), 94-8
CODEN: HCJCDB; ISSN: 0344-7138
DT Journal
LA English
CC 64-2 (Pharmaceutical Analysis)
Section cross-reference(s): 9
AB The feasibility of using **supercrit.** fluid **chromatog.** (SFC) for anal. of polar and/or ionic analytes of interest to the pharmaceutical industry is described. Specifically, the analyses of cyclosporin (a cyclic undecapeptide), several ionophores, and a group of fat-soluble vitamins are illustrated. The separation of a group of fat-soluble vitamins was evaluated on 2 bonded stationary phases, DB-5 and DB-WAX (Carbowax 20M type, ''bonded''). A new restrictor technol. known as a converging-diverging restrictor is described.
ST gas **chromatog** pharmaceutical analysis; **supercrit** fluid **chromatog** pharmaceutical analysis
IT Ionophores
(determination of, by **supercrit.** fluid **chromatog.**)
IT Pharmaceutical analysis
(**supercrit.** fluid **chromatog.** in)
IT Vitamins
RL: ANT (Analyte); ANST (Analytical study)
(fat-soluble, determination of, by **supercrit.** fluid **chromatog**.)
IT **Chromatography**, gas
(**supercrit.**, in pharmaceutical anal.)
IT **50-14-6**, **Vitamin D2** 57-87-4, Ergosterol 58-27-5, Vitamin K3 58-95-7, Vitamin E acetate **67-97-0**, **Vitamin D3** 68-26-8, Vitamin A 84-80-0, Vitamin K1 127-47-9, Vitamin A acetate 10191-41-0 59865-13-3, Cyclosporin 92883-58-4 115825-69-9
RL: ANT (Analyte); ANST (Analytical study)
(determination of, by **supercrit.** fluid **chromatog.**)
IT **50-14-6**, **Vitamin D2** **67-97-0**, **Vitamin D3**

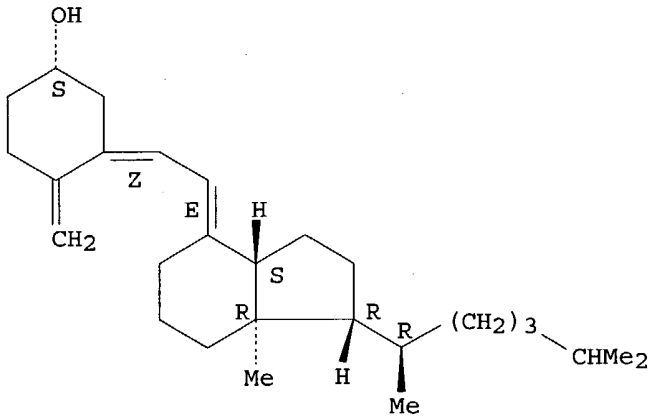
RL: ANT (Analyte); ANST (Analytical study)
(determination of, by **supercrit.** fluid **chromatog.**)
RN 50-14-6 HCAPLUS
CN 9,10-Secoergosta-5,7,10(19),22-tetraen-3-ol, (3β,5Z,7E,22E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

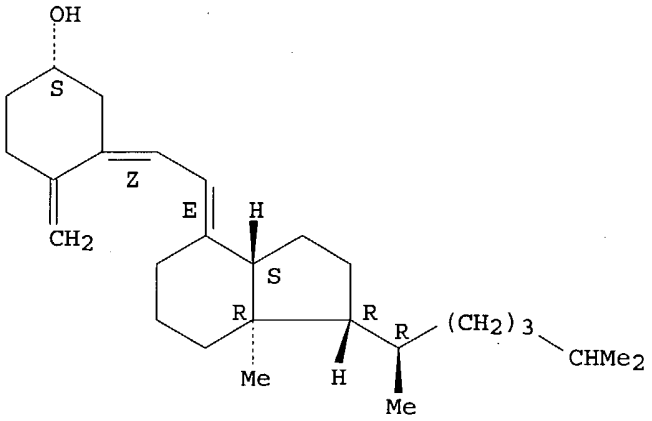
Absolute stereochemistry.
Double bond geometry as shown.



L96 ANSWER 7 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1987:81180** HCAPLUS
DN **106:81180**
ED Entered STN: 21 Mar 1987
TI Fundamental conditions in pressure-programmed **supercritical** fluid **chromatography**-mass spectrometry and some applications to vitamin analysis
AU Matsumoto, K.; Tsuge, S.; Hirata, Y.
CS Fac. Eng., Nagoya Univ., Nagoya, 464, Japan
SO Chromatographia (1986), 21(11), 617-21
CODEN: CHRGB7; ISSN: 0009-5893
DT Journal

LA English
CC 9-15 (Biochemical Methods)
Section cross-reference(s): 80
AB A combined system of pressure-programmed packed capillary **supercrit.** fluid **chromatog.**-mass spectrometry (SFC/MS) was constructed by using a self-spouting interface assisted by vacuum nebulizing. For the optimum operation of the SFC/MS system, fundamental anal. conditions such as the flow rate of the mobile phase, the pump pressure, and the composition of the mobile phase were examined The use of large packing materials indicated that the capacity factor for a sample solute is almost constant under a given pump pressure regardless of the flow rate of the **supercrit.** fluid. The SFC/MS system was applied to the anal. of both water- and fat-soluble vitamins. Both types of vitamins were clearly separated under basically the same SFC conditions. High quality mass spectra of the vitamins were obtained; selected ion monitoring traces of the vitamins are also reported as well as their UV traces.
ST **supercrit** fluid **chromatog** mass spectrometry; pressure programmed **supercrit** fluid **chromatog**; vacuum nebulizing interface **chromatog** spectrometry; vitamin **supercrit chromatog** mass spectrometry
IT Mass spectra
(of fat- and water-soluble vitamins)
IT Mass spectroscopy
(pressure-programmed **supercrit.** fluid **chromatog.** combined with, of vitamins, fundamental conditions in)
IT Vitamins
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit.** fluid **chromatog.**-mass spectrometric)
IT **Chromatography**, gas
(**supercrit.**, mass spectrometry combined with pressure-programmed, of vitamins, fundamental conditions in)
IT 50-81-7, Vitamin C, analysis 59-67-6, Nicotinic acid, analysis **67-97-0, Vitamin D3** 98-92-0, Nicotinamide 8059-24-3, Vitamin B6 11103-57-4, Vitamin A
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit.** fluid **chromatog.**-mass spectrometric)
IT **67-97-0, Vitamin D3**
RL: ANST (Analytical study)
(separation and identification of, pressure-programmed **supercrit.** fluid **chromatog.**-mass spectrometric)
RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



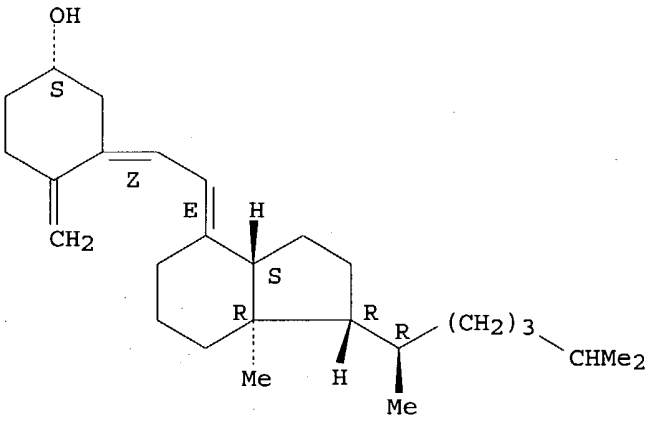
L96 ANSWER 8 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1985:106428** HCAPLUS
DN **102:106428**
ED Entered STN: 06 Apr 1985
TI Separation of hydroxylated **vitamin D3** metabolites by high-performance liquid **chromatography**
AU Moysan, J. F.; Berthou, F.; Floch, H. H.
CS Lab. Biochim., Fac. Med. Brest, Brest, 29200, Fr.
SO Pathologie Biologie (1984), 32(8), 825-7
CODEN: PTBIAN; ISSN: 0031-3009
DT Journal
LA French
CC 2-1 (Mammalian Hormones)
Section cross-reference(s): 9
AB The hydroxylated metabolites of **vitamin D3** were separated by normal-phase HPLC on Spherisorb 5SW-5, Radpak silica-10 μm, and Zorbax silica-5 μm columns and by reversed-phase HPLC on Vydac 201 TP C18-5 μm, Radpak C18-10 μm, and Ultraspher C18-5 columns. With the normal-phase **chromatog.** systems, the order of elution of the hydroxylated metabolites was 25-hydroxyvitamin **D3** and (E)-25-hydroxyvitamin **D3**, then (24R), 25-dihydroxyvitamin **D3**, 25(S), 26-dihydroxyvitamin **D3**, and 1α,25-dihydroxyvitamin **D3**. However, this order of elution was variable with the Radpak silica column as a function of the nature of the mobile phase. The level of MeOH in the mobile phase had a **critical** effect on the separation and caused the appearance of perfectly sym. peaks. With the reversed-phase **chromatog.** systems, the order of elution of the metabolites was the opposite of the above elution order. In addition, the Z and E isomers of 25-hydroxyvitamin **D3** could be separated, in contrast to adsorption **chromatog.**
ST **vitamin D3** metabolite HPLC; hydroxyvitamin **D3** metabolite HPLC; **chromatog vitamin D3** metabolite
IT **Chromatography**, column and liquid
(high-performance, **vitamin D3** hydroxlyated metabolites separation by)
IT **Chromatography**, column and liquid
(high-performance, reversed-phase, **vitamin D3** hydroxlyated metabolites separation by)
IT **67-97-0D**, hydroxylated metabolites **19356-17-3** **22350-41-0** **32222-06-3** **42737-59-7** **55721-11-4**
RL: PROC (Process)
(separation of, by HPLC)

IT 7631-86-9, biological studies 94699-20-4 94699-31-7 94699-49-7 94699-60-2
RL: BIOL (Biological study)
(**vitamin D3** hydroxylated metabolite separation by HPLC on)

IT **67-97-0D**, hydroxylated metabolites **19356-17-3 22350-41-0 32222-06-3 42737-59-7 55721-11-4**
RL: PROC (Process)
(separation of, by HPLC)

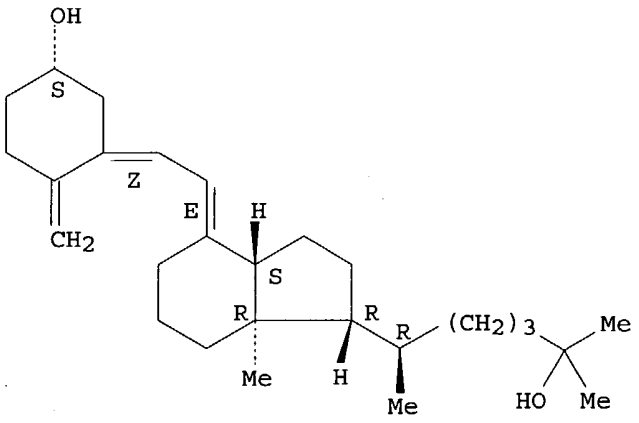
RN 67-97-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



RN 19356-17-3 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-triene-3,25-diol, (3β,5Z,7E)- (9CI) (CA INDEX NAME)

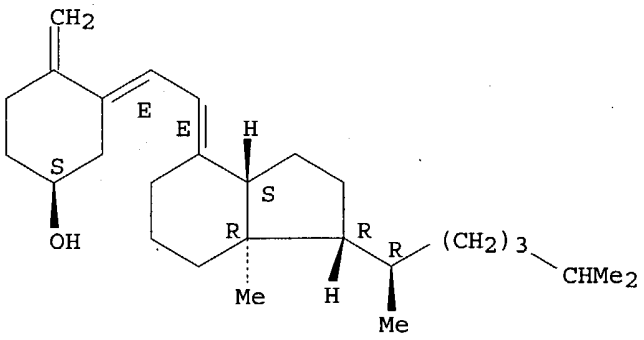
Absolute stereochemistry.
Double bond geometry as shown.



RN 22350-41-0 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-trien-3-ol, (3β,5E,7E)- (9CI) (CA INDEX NAME)

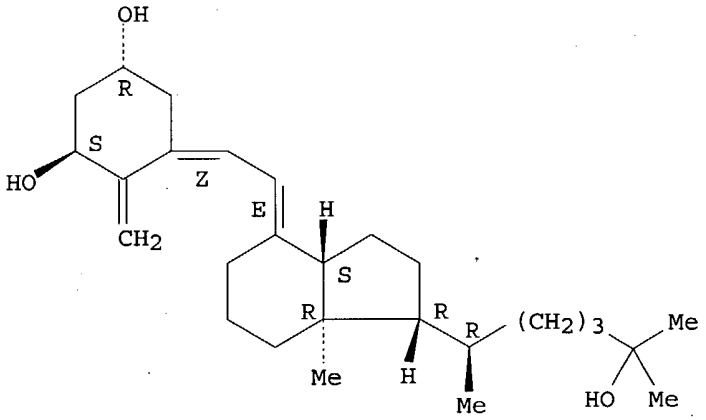
Absolute stereochemistry.

Double bond geometry as shown.



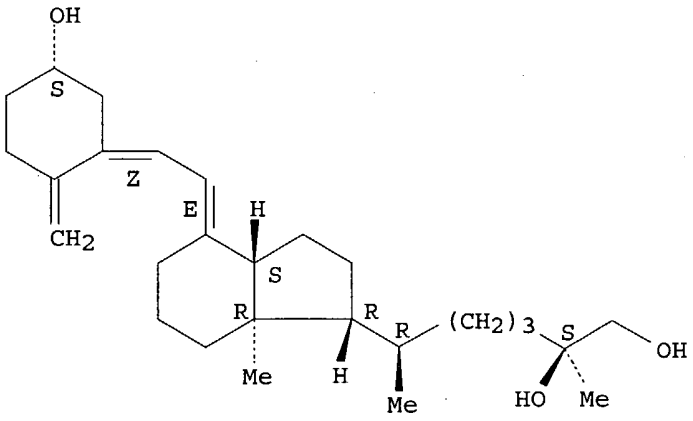
RN 32222-06-3 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-triene-1,3,25-triol, (1α,3β,5Z,7E)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



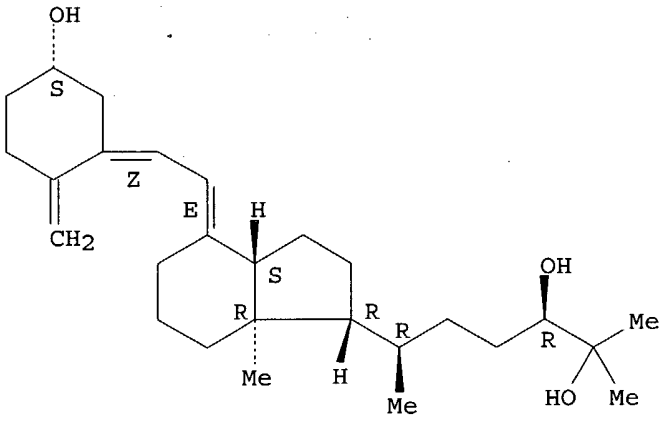
RN 42737-59-7 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-triene-3,25,26-triol, (3β,5Z,7E,25S)-
(9CI) (CA INDEX NAME)

Absolute stereochemistry.
Double bond geometry as shown.



RN 55721-11-4 HCAPLUS
CN 9,10-Secocholesta-5,7,10(19)-triene-3,24,25-triol, (3β,5Z,7E,24R)-(9CI) (CA INDEX NAME)

Absolute stereochemistry. Rotation (+).
Double bond geometry as shown.



L96 ANSWER 9 OF 9 HCAPLUS COPYRIGHT 2004 ACS on STN
AN **1967:481297** HCAPLUS
DN **67:81297**
ED Entered STN: 12 May 1984
TI Determination of **vitamin D** and of ergosterol in fodder yeasts. I. **Critical** investigation of the test methods in model solutions
AU Spanyar, Pal; Blazovich, Marta; Gabor, Mrs. Istvan
CS Kozponti Elemiszeripari Kutatoint., Budapest, Hung.
SO Elelmiszervizsgalati Kozlemenyek (1967), 13(2), 77-91
CODEN: EMKZAH; ISSN: 0422-9576
DT Journal
LA Hungarian
CC 17 (Foods)
AB A com. ergosterol (I) preparation was used to test estimation procedures. Good results were obtained by the Liebermann-Burchard color reaction of I with Ac2O and H2SO4, as well as by determining the uv absorption of the complex of I and digitonin. Procedures for **vitamin D** (II) were tested on a com. II preparation The uv absorption of II itself as well as color reactions with SbCl3 and with furfural were suitable. In the presence of each other, I and II can be determined by thin-layer **chromatog.** on **silica gel** G, using 1:1 cyclohexaneether and developing with SbCl3. 63 references.
ST **VITAMIN D** DETN YEAST; ERGOSTEROL DETN YEAST; YEAST VITAMINS DETN; FODDER YEAST VITAMINS DETN; SPANYAR P; BLAZOVICH M; GABOR I MRS
IT 57-87-4 **1406-16-2, Vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(determination of)
IT **1406-16-2, Vitamin D**
RL: ANT (Analyte); ANST (Analytical study)
(determination of)
RN 1406-16-2 HCAPLUS
CN Vitamin D (8CI, 9CI) (CA INDEX NAME)

*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***

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     (FILE 'HOME' ENTERED AT 14:46:34 ON 08 MAY 2004)
               SET COST OFF

     FILE 'REGISTRY' ENTERED AT 14:46:51 ON 08 MAY 2004
               E VITAMIN D3/CN
L1           1 S E3
               E PREVITAMIN D3/CN
L2           1 S E3
               E VITAMIN D/CN
L3           1 S E3
L4             STR
L5          50 S L4
L6        8414 S L4 FUL
               SAV TEMP L6 QAZI335/A
L7             STR L4
L8           0 S L7
L9         662 S L7 FUL
               SAV L9 TEMP QAZI335A/A
L10       9076 S L6,L9
L11       9074 S L10 NOT L1,L2
L12        366 S L11 AND ?VITAMIN?/CNS
L13       8708 S L11 NOT L12
L14          1 S CARBON DIOXIDE/CN

     FILE 'HCAPLUS' ENTERED AT 14:52:16 ON 08 MAY 2004
L15       5267 S L1
L16        235 S L2
L17      12688 S (VIT OR VITAMIN)(L)D3
L18        136 S (VIT OR VITAMIN)(L)D 3
L19       2247 S CHOLECALCIFEROL#
L20         14 S COLECALCIFEROL#
L21        249 S (PREVIT OR PREVITAMIN OR PRE()(VIT OR VITAMIN))(L)D3
L22          6 S (PREVIT OR PREVITAMIN OR PRE()(VIT OR VITAMIN))(L)D 3
L23         83 S PRECALCIFEROL# OR PRE CALCIFEROL#
L24        125 S PREVITAMIN D
L25      10602 S L3
L26      29557 S VITAMIN D#
L27      16535 S L12
L28       3123 S L13
L29      36305 S L15-L28
               E JOHANNSEN M/AU
L30         17 S E3,E8
L31          2 S L30 AND L29
L32        270 S L29 AND (LAROCHE? OR LA ROCHE? OR HOFFMANN?)/PA,CS
L33          1 S US20010001801/PN OR EP98-111490/AP,PRN
L34     176984 S L14
L35     441586 S CARBON DIOXIDE OR CO2
L36        204 S CARBONDIOXIDE
L37        184 S L29 AND L34-L36
L38          4 S L37 AND L31,L32,L33
               E CHROMATOGRAPHY/CT
L39          1 S L37 AND E30
L40          3 S L37 AND E110
L41          2 S L37 AND E145,E151,E154
L42          0 S L37 AND E158
L43          2 S L37 AND E27-E29
               E E3+ALL
L44          2 S L37 AND E4-E6
L45         12 S L37 AND E3+NT
               E SILICA GEL/CT
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L46           2 S L37 AND E4-E28
                E E3+ALL
L47           2 S L37 AND E16,E15+NT
L48          18 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT?)
L49           1 S L37 AND FLASH?
L50          23 S L33,L38-L49
                E SEPARATION/CT
L51         561 S E3+OLD,NT,PFT AND L29
                E PURIFICATION/CT
L52           9 S E3+OLD,NT,PFT AND L29
                E ISOLATION/CT
L53         561 S L51-L52
L54          19 S L37 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
L55           0 S L37 AND (BACKPRESSUR? OR BACK PRESSUR?)
L56          22 S L37 AND ?CHROMATOG?
L57          30 S L54,L56,L50
L58          20 S L57 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L59         473 S (PROVIT? OR PRO VIT?)()D#
L60         172 S L59 NOT L29
L61         163 S L60 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L62          23 S L61 AND ?CHROMATO?
L63           0 S L61 AND (BACKPRESSUR? OR BACK PRESSUR?)
L64           0 S L61 AND (?SUPERCRIT? OR ?SUPER CRIT? OR FLASH?)
L65           7 S L61 AND L34-L36
L66           2 S L61 AND CHROMATOGRAPHY+OLD,NT,PFT/CT
L67           2 S L61 AND SEPARATION+OLD,NT,PFT/CT
L68           0 S L61 AND PURIFICATION+OLD,NT,PFT/CT
L69          49 S L62,L65-L67,L58
L70          49 S L69 AND (PY<=1999 OR PRY<=1999 OR AY<=1999)
L71          10 S L57 NOT L70
L72           8 S L70 AND ?CRITIC?
L73          27 S L70 AND L34-L36
L74          27 S L72,L73
L75          22 S L70 NOT L74
                SEL DN AN L74 25
L76          26 S L74 NOT E1-E3
L77          36 S L71,L76
L78          12 S L77 AND (?RADIAT? OR UV OR ULTRAVIOL? OR ULTRA VIOL?)
L79          36 S L77,L78

     FILE 'REGISTRY' ENTERED AT 15:17:03 ON 08 MAY 2004

     FILE 'HCAPLUS' ENTERED AT 15:18:23 ON 08 MAY 2004
L80       36477 S L29,L59
L81         471 S L80 AND ?CRITIC?
L82          28 S L80 AND ?SUPERCRITIC?
L83           0 S L80 AND ?SUPER CRITIC?
L84           0 S L80 AND ?ULTRACRITIC?
L85          36 S L81 AND ?CHROMATOG?
L86          44 S L82,L85
L87          21 S L81 AND L34-L36
L88          29 S L86,L87 NOT L79
                SEL DN AN 6 9-12 14-17 19 22 23 24 26
L89          14 S L88 AND E4-E45
L90           2 S L89 AND SILICA GEL
L91          14 S L89,L89
L92           7 S L91 AND L82
L93           8 S L90,L92
L94           6 S L91 NOT L93
                SEL DN AN 2
L95           1 S E46-E48 AND L94
L96           9 S L93,L95
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